METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 61 No. 367 MAY, 1960 Monthly: Two Shillings and Sixpence

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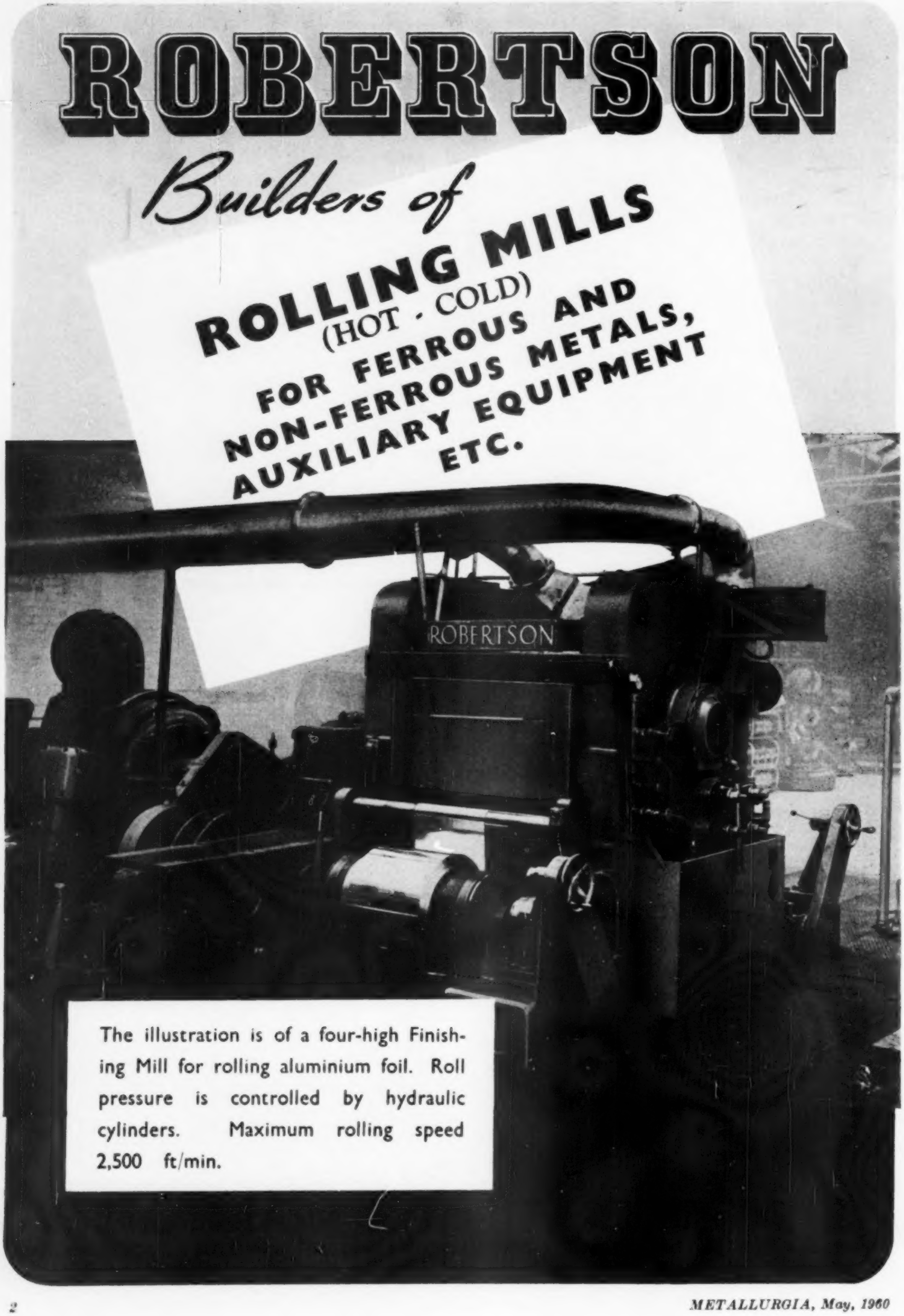

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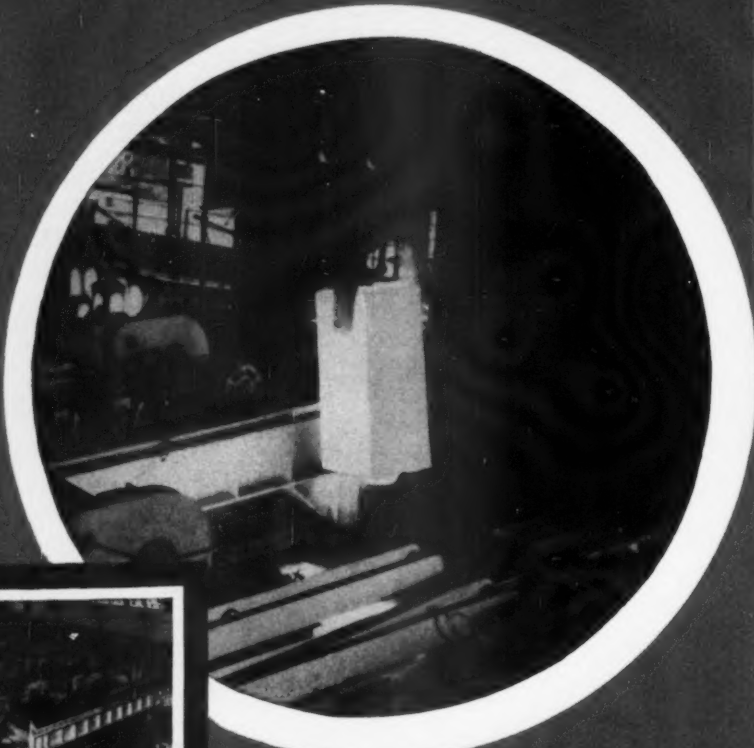

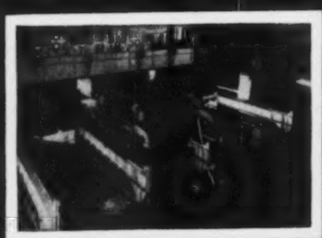

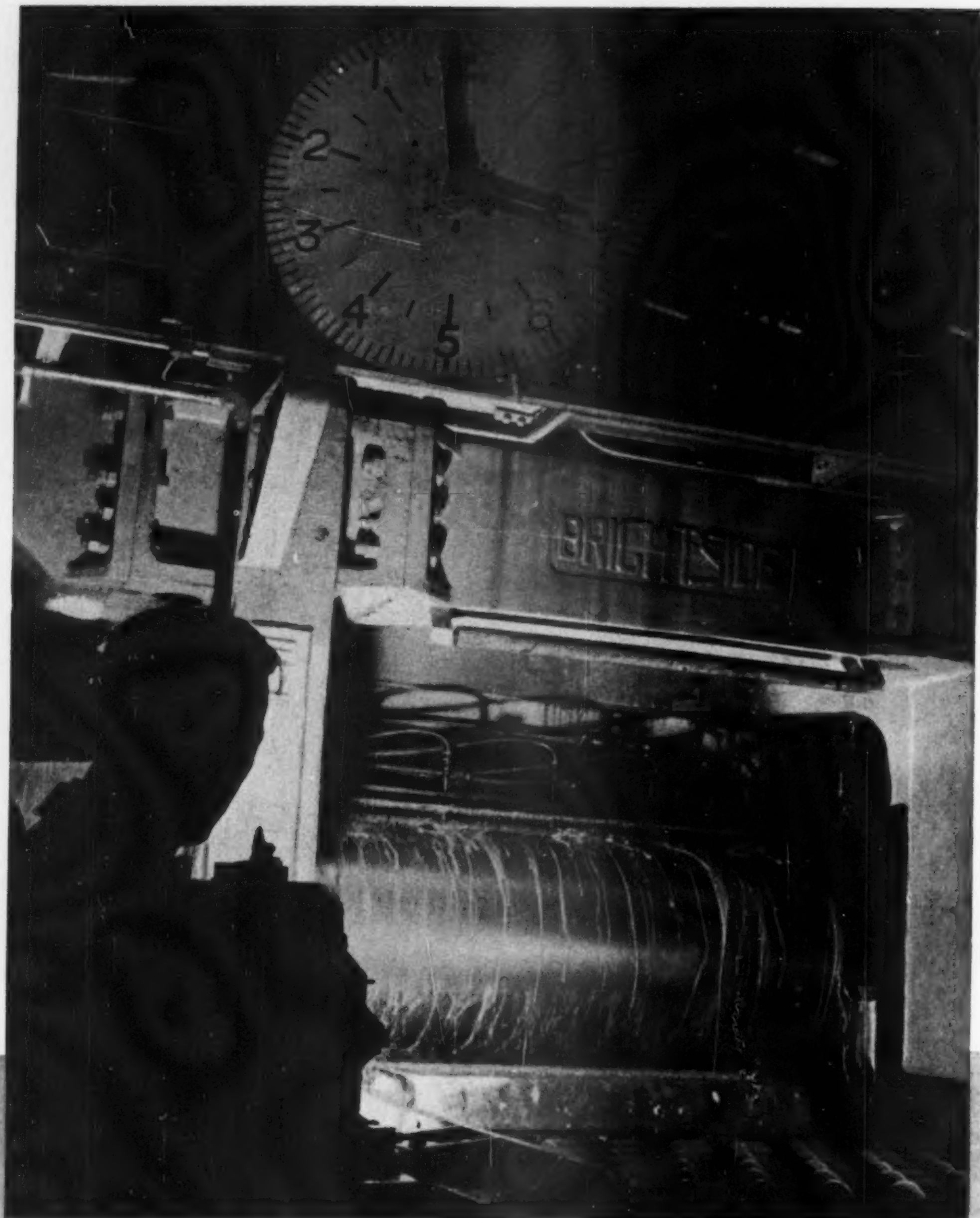
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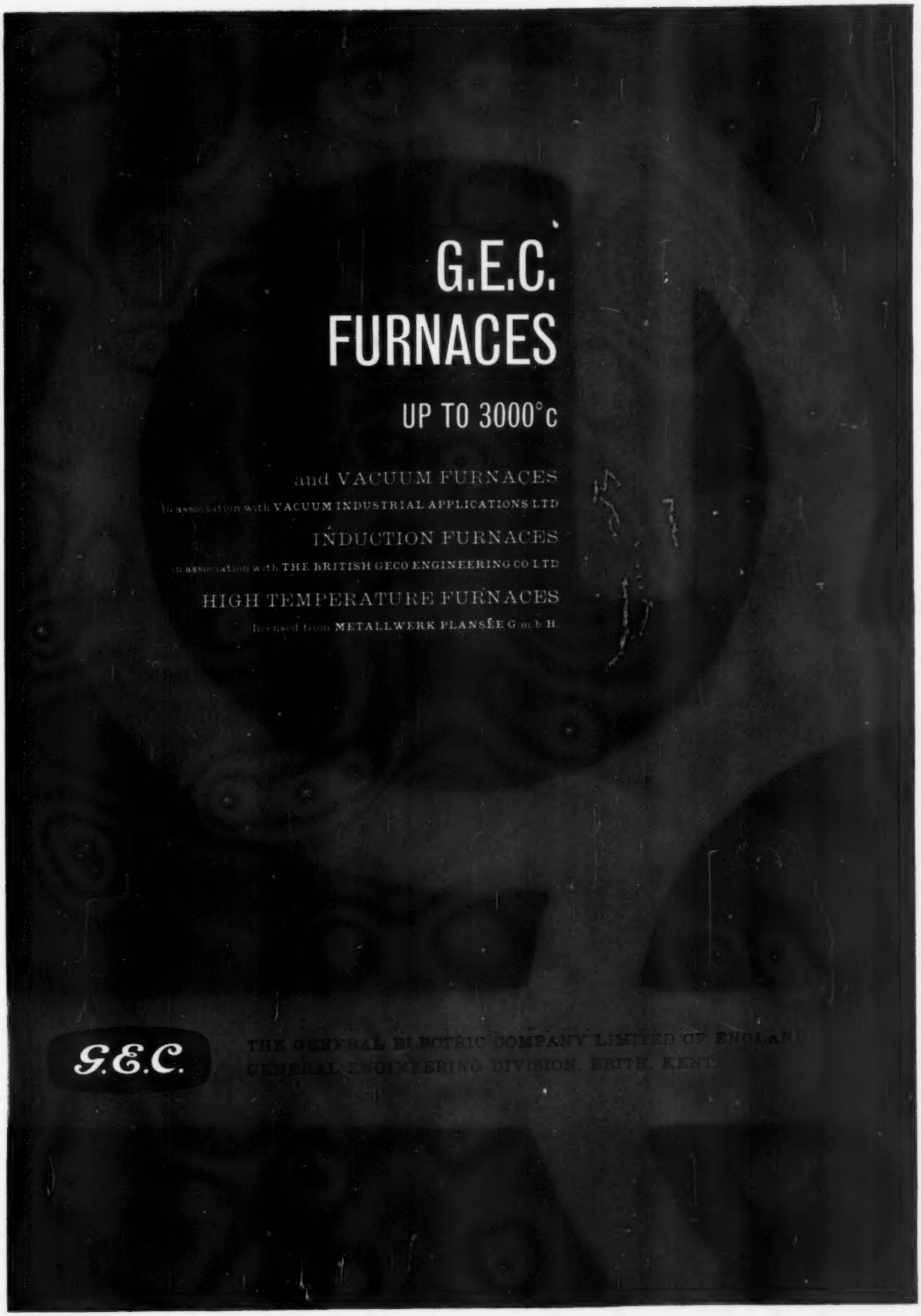
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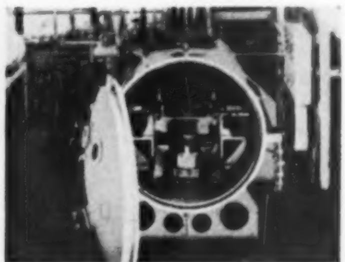

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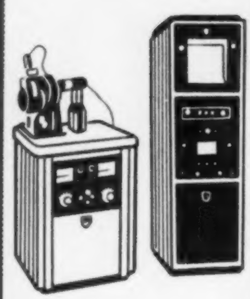

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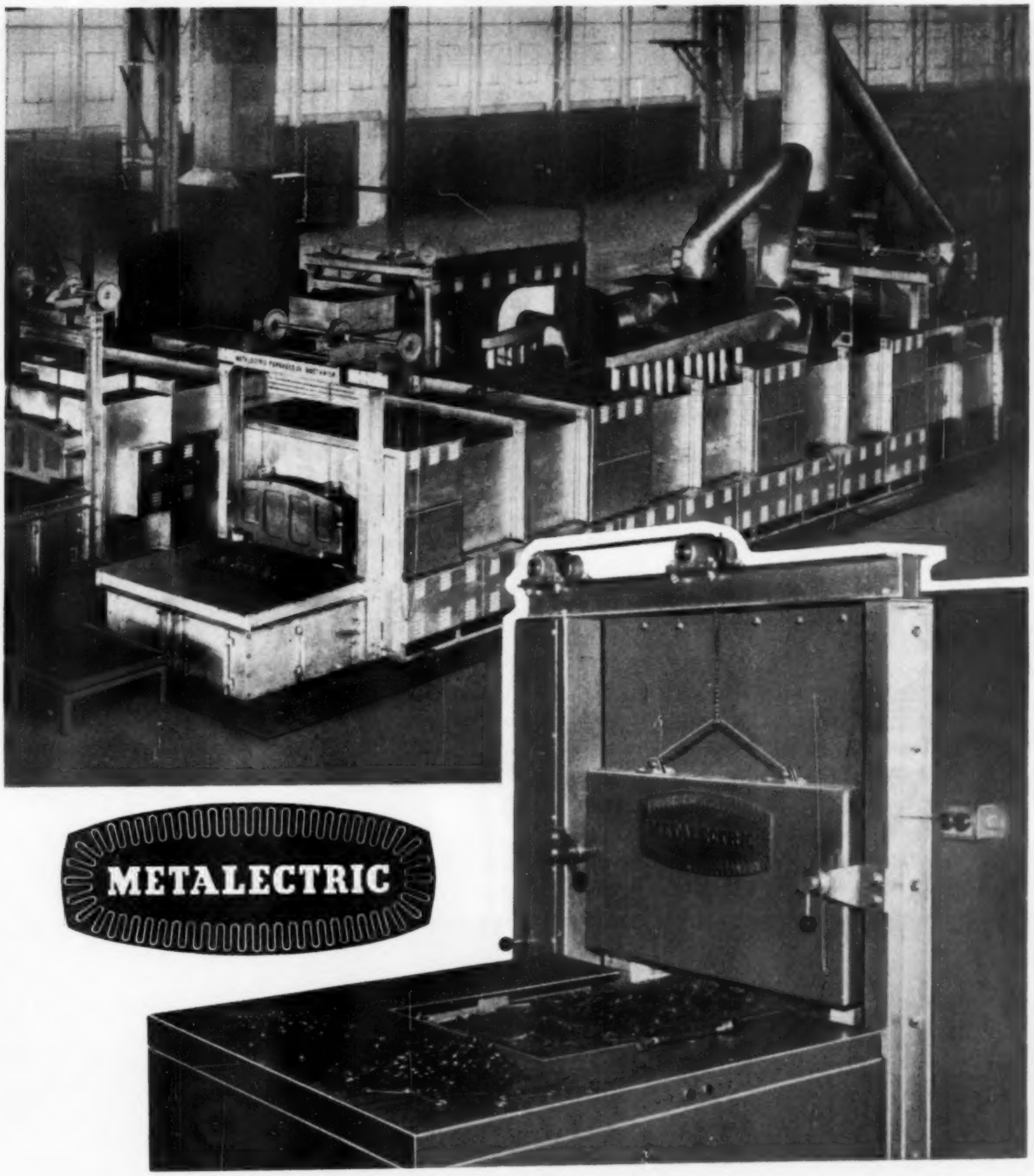

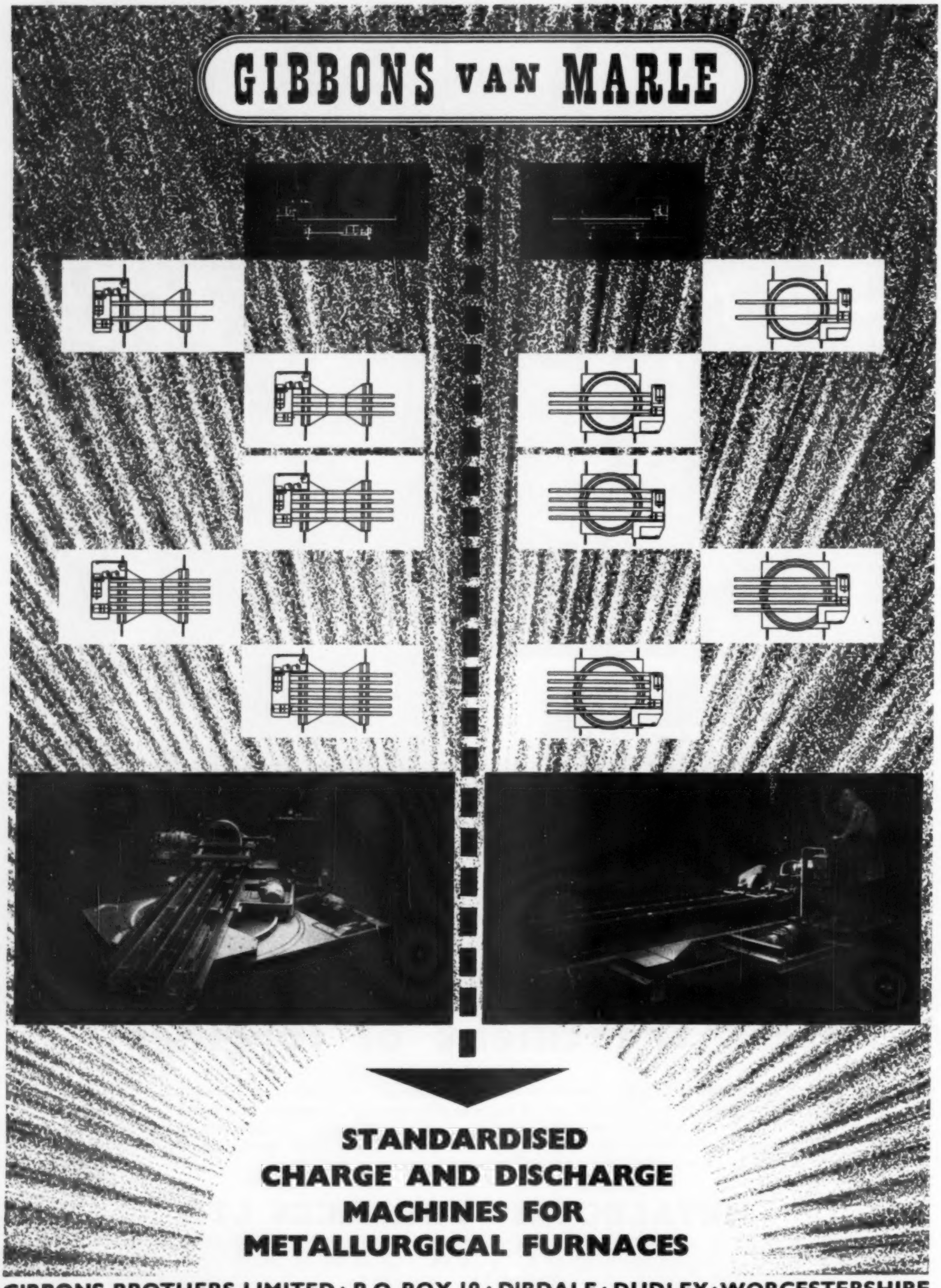
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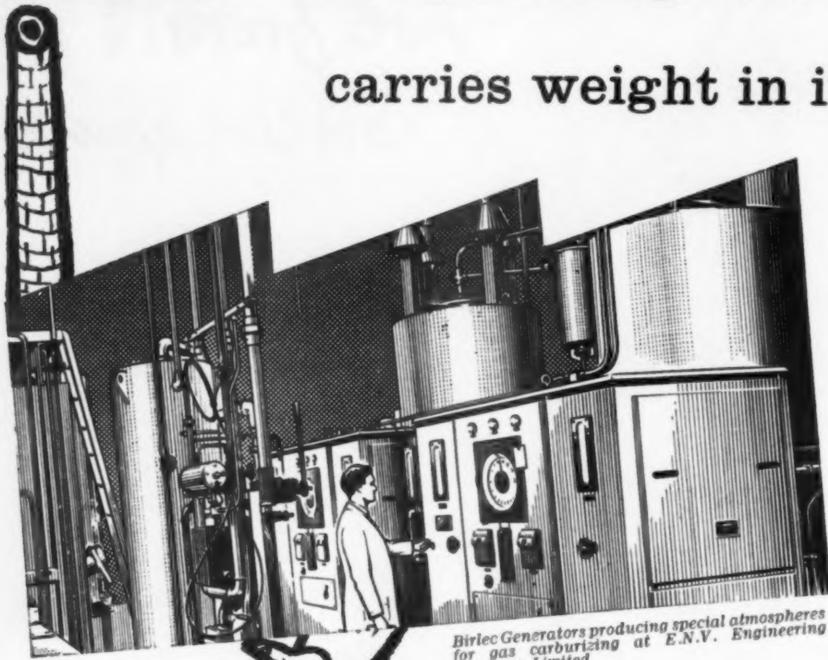

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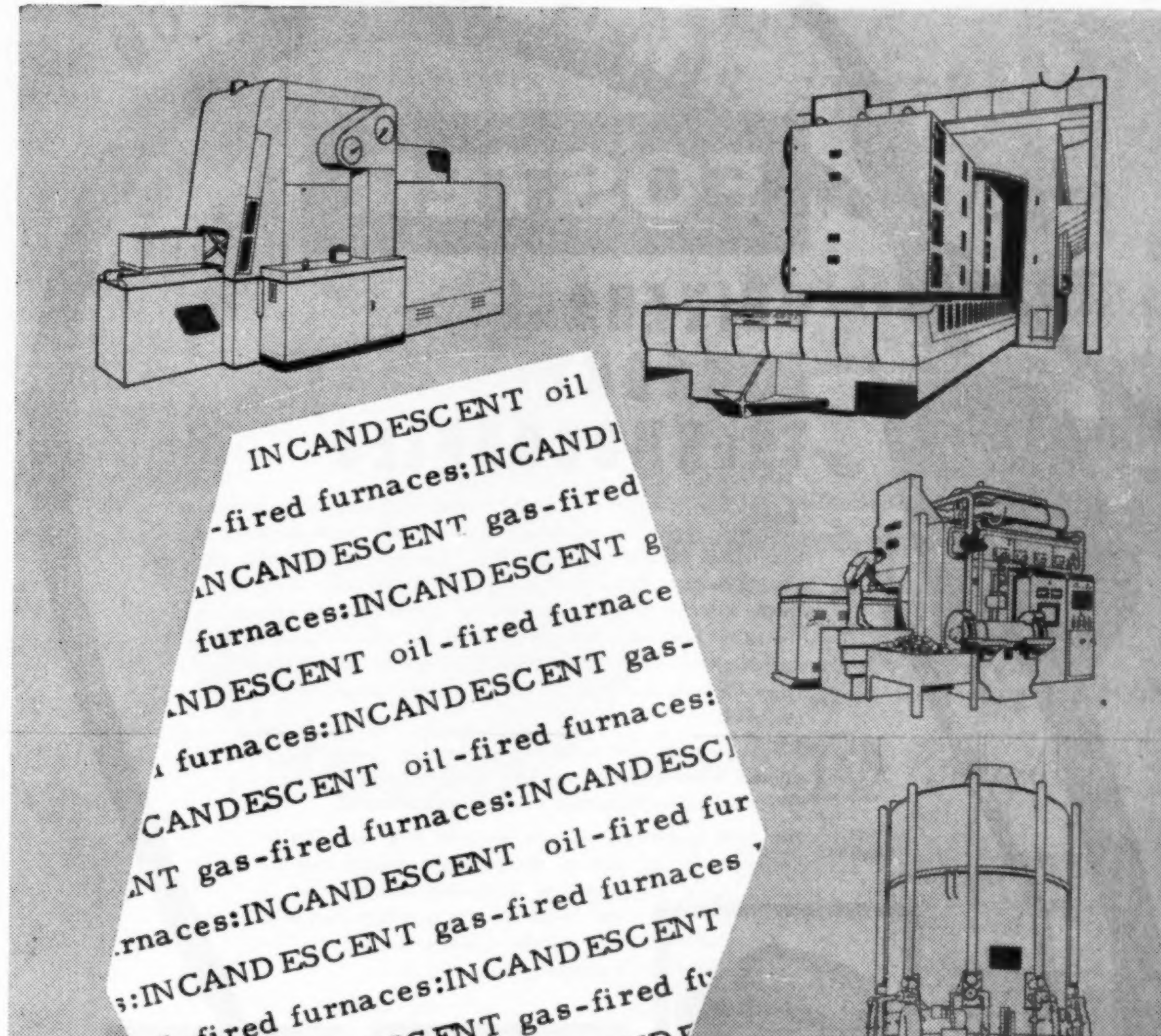
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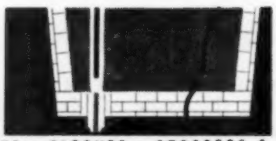

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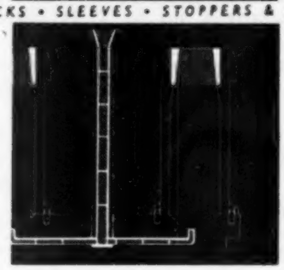

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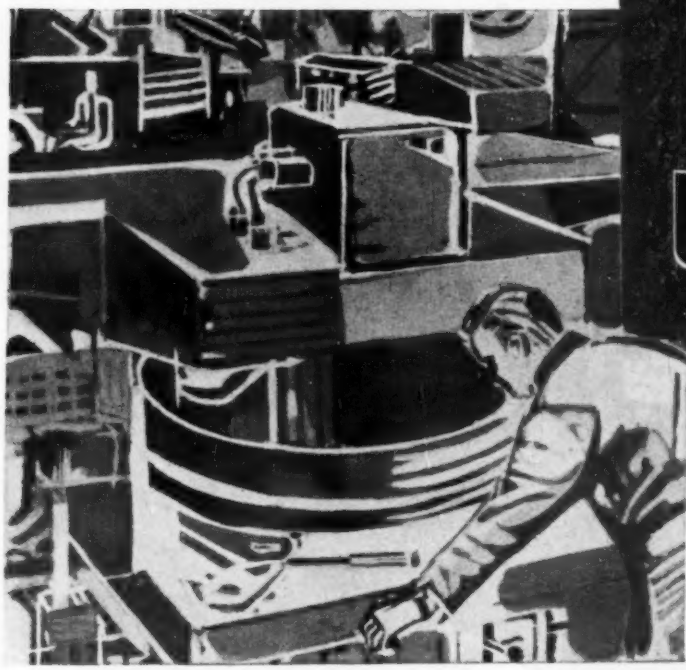

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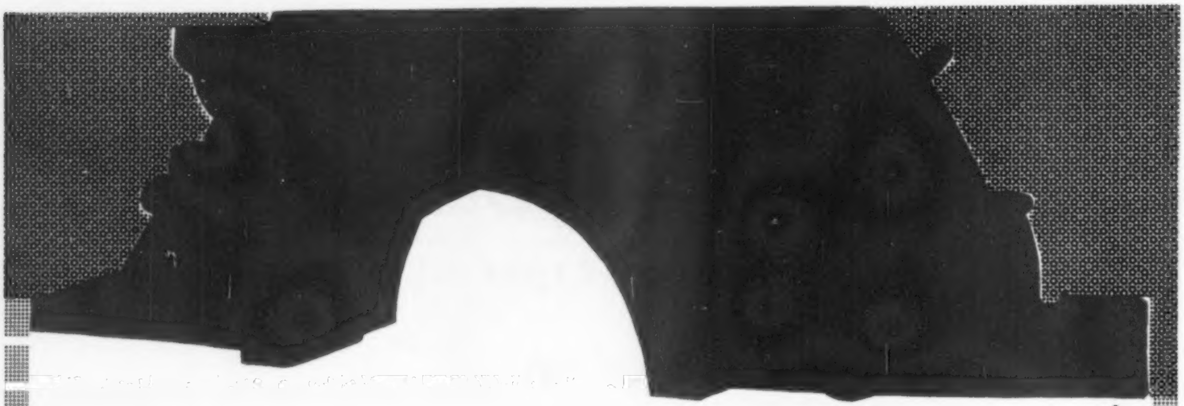

METALLURGIA

THE BRITISH JOURNAL OF METALS
INCORPORATING THE METALLURGICAL ENGINEER

CONTENTS FOR MAY, 1960 Vol. 61 No. 367.

PUBLISHED MONTHLY BY

The Kennedy Press, Ltd.,
31, King Street West,
Manchester, 3.
Telephone: BLAckfriars 2084.

London Office:

158, Temple Chambers,
Temple Avenue, E.C.4.
FLEet Street 8914.

CONTRIBUTIONS

Readers are invited to submit articles for publication in the editorial pages: photographs and/or drawings suitable for reproduction are especially welcome. Contributions are paid for at the usual rates. We accept no responsibility in connection with submitted manuscript. All editorial communications should be addressed to The Editor, "Metallurgia," 31, King Street West, Manchester, 3.

SUBSCRIPTIONS

Subscription Rates throughout the World—30/- per annum, Post free.

ADVERTISING

Communications and enquiries should be addressed to the Advertisement Manager at Manchester.

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METALLURGIA

THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER"

May, 1960 Vol. LXI. No. 367

European Steel Trends

LOOKING into the future is a fascinating, if somewhat risky, business. The risks involved have not been underestimated by the ECE Steel Committee in its attempts to ascertain, first, the likely trend of long-term requirements of steel products in Europe and the rest of the world for the next fifteen years; secondly, the likely evolution, during this period, of iron and steel production in Europe and overseas, against the background of technological development in the recent past and to be expected in the future, and its influence on investment in the iron and steel industry and on the growth of productivity; thirdly, the adequacy or otherwise of the likely future supply of steelmaking raw materials in the light of probable levels of steel production and of possible trade in these materials; and fourthly, an assessment of Europe's direct export prospects by products and, if possible, of her indirect export prospects (i.e., in the form of goods made from steel). Nevertheless, it is the hope of the Committee that the report* containing its findings will prove a valuable contribution to an assessment of the future prospects of the European steel industry; at the least, the comprehensive statistics which have been assembled should be of value to these wishing to make their own appraisal of the situation.

Steel consuming industries today fall into three distinct groups with *per capita* steel consumptions of: less than 5 kg.; 5–50 kg.; and more than 90–100 kg.; and it is rather surprising to learn that the experience of recent decades has shown that few countries are able to move steadily from lower to higher levels and thus pass into a different group: this is attributed to marked disturbances in normal economic growth through two world wars and the depression of the early 'thirties.

During the early stages of economic development, steel is mainly used for investment, but as growth proceeds an increasing share is directed to consumption in various forms. Later, the shares of production and transport equipment in the investment sector tend to increase at the expense of construction. These then start to decline again and construction increases. This general pattern is obviously strongly affected by specific economic specialisations of particular countries, and the distribution of steel among sectors of utilisation is markedly influenced in many countries by indirect trade.

In estimating the future fifteen years ahead, the Committee has had to take into account the tendency for the rate of increase of steel consumption to fall as the absolute value rises and to assume that economic growth will be smooth, and that economic development will either start or continue in most under-developed regions or countries. Within the framework of these principles and assumptions, estimates have been derived for the world as a whole and for regions and countries, and then adjusted by over-all economic evaluation. It is expected that the share of industrial flat products will increase, although not uniformly from region to region. This is particularly true of tinplate, except that there may be a relative decline in regions at present specialising in agriculture and canning. A decrease in the share of sections and railway track material is likely, except in the Far East, and a relatively slow decline in the share of steel tubes can be expected in the oil-producing regions of Latin America and the Middle East; in other regions the share of tubes seems likely to grow. It is difficult to assess with any great accuracy the product pattern of steel consumption fifteen years hence due to gaps in the information available. Furthermore there is the possibility, in the years ahead, of the substitution of steel by other materials, particularly plastics and light metals, in certain sectors of utilisation. Again, in industries such as building and civil engineering, accounting for about a quarter of total steel consumption, technical development is at present fairly rapid and, consequently, there are changing patterns in the use of materials.

Foreign trade in steel may be classed as "deficit-covering" and "exchange," and although both have grown, the latter has increased more rapidly than the former, indicating a trend towards greater self-sufficiency in steel. Estimates show an increase in exchange in the next fifteen years from 10·4 million to 18·9 million tons, and in deficit-covering from 20·3 million to 28·4 million tons. It is estimated that indirect trade will increase from 16·7 million to 38·0 million tons, so that the ratio of indirect to direct trade will increase. Europe accounts for some 50% of world exports, excluding intra-European trade, and a recent slight downward trend has been offset by higher value exports and growing indirect exports, also of higher value. Price, quality and delivery, as factors accounting for relative shares of export markets, have so far been of secondary importance, but the situation may change, and a study of the position should, in the Committee's view, prove rewarding.

The availability of raw materials to meet the demands of increased steel production will be governed by a number of factors. Until the middle sixties there will be a need to increase blast furnace capacity as quickly as possible, but later increased quantities of capital scrap will be available. Improved ore preparation has led to a significant reduction in specific coke consumption, and the Committee considers that there are not likely to be any serious shortages of coke. Iron ore, too, should be plentifully available, but Europe will have to depend increasingly on overseas sources of supply in Africa and North and South America. The quality may be poorer, however, and increasing resort to beneficiation will be necessary.

* "Long-term Trends and Problems of the European Steel Industry" a United Nations publication prepared by the Secretariat of the Economic Commission for Europe. Available from H.M. Stationery Office, price 14s.

The size of blast furnaces is expected to increase over the next fifteen years to 2,000–2,500 cu.m., with outputs of 4,000–5,000 tons/day. At the same time, it is forecast that the heat-sizes of steel-making units will increase to 900–1,000 tons for open hearth furnaces; 250–300 tons for electric furnaces; and 200–300 tons for converters. The converter and electric furnace are likely to share an increasing proportion of total steel output. Further developments, which are not yet fully established, but could in the long run substantially reduce investment costs, are continuous casting and the direct reduction of ores.

In concluding its report, the Committee refers to the basic economic premise underlying the study, namely the conception of decreasing rates of steel consumption; and points out that the real implication is that the consumption and production of any one material is not an end in itself. As in the past, the possibility exists that consumption of other basic materials is likely, sooner or later, to grow to such an extent as to make further increases in steel consumption unnecessary. In the meantime, the next fifteen years should see a large absolute increase in world steel consumption and production, if the pace of general economic development which can be foreseen, and which is necessary to improve living standards everywhere, is to be realised.

Personal News

Dr. R. L. P. Berry, beryllium project manager since 1958, has been appointed to the board of I.C.I. Metals Division. Dr. Berry, who is 41, has also been appointed to the delegate boards of two I.C.I. subsidiary companies, Marston Excelsior, Ltd. and Lightning Fasteners, Ltd.

Mr. R. S. H. Shepard, M.C., T.D., and Mr. L. B. Devins have been appointed joint managing directors of The Sheffield Wire Rope Co., Ltd., a member of the Firth Cleveland Group. Mr Shepard was formerly a director and Mr. Devins an executive director and general manager.

Mr. I. A. Bailey has been appointed deputy chairman of The Mond Nickel Co., Ltd., and of Henry Wiggin & Co., Ltd., and Mr. E. Vaughan has been appointed a director of the two companies.

Mr. R. P. Janion has been appointed sales engineer of the switchgear division at the Witton Works of The General Electric Co., Ltd., responsible for switchgear for voltages of 3·3 kV. and above.

Edgar Allen & Co., Ltd., of Sheffield have announced the appointment of Mr. W. H. Everard as deputy general manager of the foundry division, with Mr. J. M. T. Levesley as his assistant.

Mr. D. R. Mackay has been appointed regional manager of I.C.I. Midland sales region in succession to Mr. T. H. Minton, who retired on 31st March after thirty-eight years service with I.C.I. and its predecessors. Mr. Mackay, has been regional manager of the company's Scotland and Northern Ireland sales region since 1957, in which position he is succeeded by Dr. J.P. Dickson, who has been assistant I.C.I. sales controller in London since 1958.

Mr. R. Walker has recently joined Kelvin & Hughes (Industrial), Ltd., as a specialist representative for North Wales, the East and West Ridings of Yorkshire and the northern parts of Lincolnshire and Derbyshire. Previous to joining Kelvin Hughes, Mr. Walker was a metallurgist with the U.K.A.E.A. at Windscale.

Mr. E. N. Griffith, (chairman and joint managing director of Rotary Hoes, Ltd.) has been elected vice-president of the British Engineers' Association after having served on the governing council for the past ten years as the representative of the Agricultural Engineers Association.

Mr. J. R. Cotterill has been appointed manager of the outside services department at the Witton Works of The General Electric Co., Ltd. This is a new department formed to co-ordinate erection, commissioning, and post-commissioning service of electrical plant.

Mr. G. Liley has been appointed to the Board of British Lead Mills, Ltd., a member of the Firth Cleveland Group. Mr. Liley was formerly an executive director of the company.

In recognition of his outstanding services to the British gear-manufacturing industry, Mr. A. Sykes, technical consultant to the David Brown Gear Group was recently presented with a gold medal by the British Gear Manufacturers' Association. Mr. Sykes first joined the company in 1905, and prior to his present appointment he had successively served as chief engineer, works manager and engineering controller. Outside the company Mr. Sykes has a long record of service on committees concerned with gear-making and production engineering.

Mr. C. Pullan, sales director of Armstrong Whitworth (Metal Industries), Ltd., was recently presented with a gold watch to mark his retirement from executive duties after thirty years' association with the company. Certain changes in the administrative structure have been made as a result of Mr. Pullan's retirement. Mr. F. L. Turnbull, formerly director and works manager, now becomes director and general manager; Mr. R. H. Philip, formerly director and secretary, is now appointed commercial director and secretary.

Mr. H. S. Wingate, president of The International Nickel Co. of Canada, Ltd., since 1954, and a director since 1942, has been elected chairman of the board and chief officer of the company. He is succeeded as president by Mr. J. R. Gordon, who has been executive vice-president since 1957, and a director since 1953. Mr. R. D. Parker, a director since 1957, and vice-president in charge of Canadian operations since 1955, is now senior vice-president of the company. Mr. Wingate succeeds Dr. J. F. Thompson, who retires as chairman after having served the company for fifty-three years, and as chairman of the board and chief officer since 1951. Dr. Thompson becomes honorary chairman and will continue to serve as chairman of the executive committee. The board of directors of the company's United States subsidiary, The International Nickel Company, Inc., have elected Mr. Wingate chairman, Mr. Gordon president, and Dr. Thompson honorary chairman and chairman of the executive committee of that company.

Mr. R. D. Pearce has been appointed a director of Keeton, Sons & Co., Ltd., a member of the Firth Cleveland Group.

Some Characteristics of Integron Mild Steel Tubing for Heat Exchangers in Nuclear Power Stations

By F. E. Asbury, B.Met., A.I.M. and L. H. Toft, B.Sc., A.I.M.

Central Electricity Research Laboratories

Consequent upon the decision to use Integron mild steel tubing for the heat exchangers of the Berkeley and Bradwell nuclear power stations, the following aspects of the characteristics and behaviour of this material have been investigated : the general metallurgical characteristics and the fatigue resistance of the tubing in the " as-finned " and in the heat treated conditions ; the effect of service temperature conditions on the tensile properties and residual stress ; and the effect of welding on the parent material. The results of these investigations show that mild steel Integron tubing should be satisfactory for service without heat treating under the conditions proposed for the Bradwell and Berkeley heat exchangers.

INTEGRON mild steel tubing is being manufactured by Imperial Chemical Industries, Ltd., for the heat exchangers of the Berkeley and Bradwell nuclear power stations. In the Integron process, the outer surface of plain tubing is cold rolled to produce circumferential fins, and the material comes from the mill in a stressed and work hardened condition.

The investigations described in this paper were undertaken to reveal any significant effects that might arise from the fin root profile imparted by the Integron process, or from the use of the finned tubing in the " as-finned " condition as compared with heat treated conditions. Plain unfinned mild steel boiler tubing is used in an annealed or normalised condition to facilitate cold forming operations, and in the softened condition the optimum creep properties are probably obtained. The Integron tubing will not be subjected to further cold forming in the heat exchanger designs for Berkeley and Bradwell, and the conditions of service are far below the creep capacity of the material. It was, therefore, considered that it would be unnecessary to heat treat the Integron tubing if the behaviour in other respects was shown to be satisfactory.

The Integron tubing for Bradwell has a nominal internal diameter of 1⅝ in. and is rolled from seamless hot-rolled tubing. The tubing for Berkeley is nominally ⅞ in. internal diameter, and is rolled from electric resistance-welded tube (ERW). Fully aluminium killed fine grained mild steel has been specified for Integron tubing to be used for nuclear power station heat exchangers to minimise strain age-hardening effects arising from the cold work imparted by the finning process.

The finned tubes will be used in the evaporator and economiser sections of the heat exchangers, and will carry mixtures of water and steam at temperatures in the range 170°–260° C. Carbon dioxide gas from the reactors will pass over the outside of the tubes. The temperatures reached by the Integron tubing in normal operation will lie in the range 200°–320° C., and cannot under any conditions exceed the temperature of the carbon dioxide, 390° C. at Bradwell and 345° C. at Berkeley. The stresses due to the excess internal pressure of the steam-water mixture, over the external pressure of the carbon dioxide gas, will be well below the capacity of the material.

Three aspects of the characteristics and behaviour of mild steel Integron were investigated at the Central Electricity Research Laboratories. They were :—

(1) The general metallurgical characteristics and the

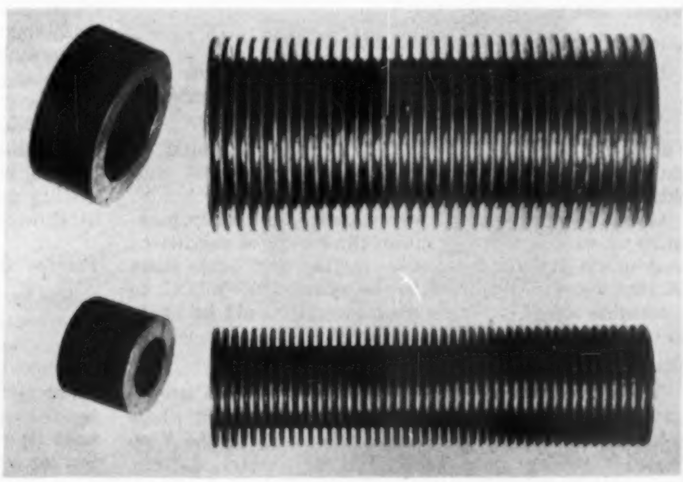


Fig. 1.—Integron finned mild steel tubes with parent tubing on left. Top : Bradwell size ; bottom : Berkeley size.

× ½ approx.

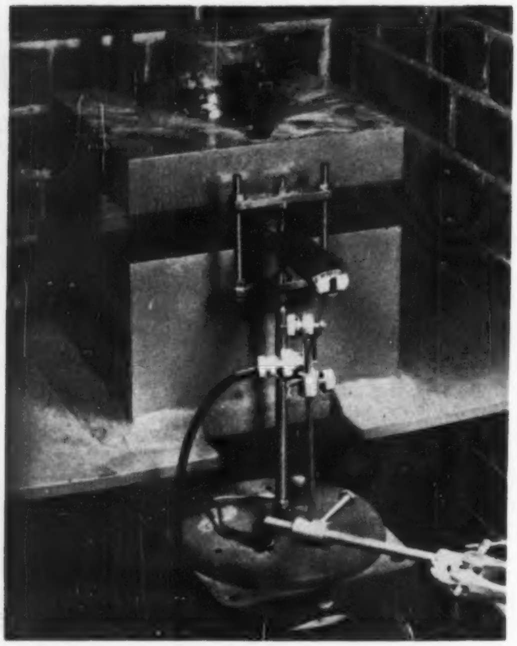


Fig. 2.—Vibration rig with Integron specimen under test.

fatigue resistance of the tubing in the " as-finned " and in the heat treated conditions.

(2) The effect of service temperature conditions on the tensile properties and residual stresses.

(3) The effect of welding on the parent material.

Experimental Procedures

Samples of both the Berkeley and the Bradwell tubing were examined for external features, microstructure and hardness. The examinations involving heat treatment—including welding and the fatigue tests—were carried out only on the Integron for Bradwell, but the characteristics of the two types of tubing were not expected to differ in any important respect.

Some of the Bradwell tubing was heat treated under the following two conditions to provide material for fatigue and tensile tests :—

(*a*) Sub-critical anneal—625° C. in air.

(*b*) Normalising treatment—920° C. packed in container with cast iron borings, and the container with contents air cooled.

These treatments are typical of those that would be employed in production, if heat treatment was considered necessary.

Other heat treatments were carried out at temperatures up to 450° C. to simulate the effects of conditions that might be encountered in service, and some short lengths were heat treated in the range 625°–700° C. to determine whether severe grain growth would be likely to occur during sub-critical annealing.

General and Metallographic Examination

The dimensions of the tubing, minor surface irregularities, and the fin profiles were investigated on whole tubes or sections of the tubing. Sections of the " as-finned " tubing were examined for surface defects, inclusions, grain size, and work hardening, whilst sections of the heat treated material were examined for re-crystallisation.

Tensile and Hardness Tests

The tensile properties of specimens from finned tubes in the normalised and " as-finned " conditions were determined at temperatures up to 450° C., to assess the extent of strain age-hardening effects.

Room temperature tensile tests and hardness tests were carried out on specimens machined from finned tubes, which had been heat treated for 24 hours at temperatures up to 450° C., and on specimens machined from normalised and sub-critically annealed (625° C.) tubes.

Hounsfield specimens, machined longitudinally from the centre of the tube wall, were used for the tensile tests, and therefore the heavily cold worked outer layers were not included in the constricted part of each test-piece.

Hardness surveys were made on longitudinal sections of the tube in the upper part of the fins, and between the base of the grooves and the bore of the tube.

Residual Stresses

The Integron rolling procedure was expected to leave high residual stresses in the tubing. These stresses could lower the resistance of the Integron tubing to distortion during, for example, handling, by causing plastic flow at relatively low applied loads, and they might also cause the Integron to distort when machined at points of attachment. In addition, the fatigue strength of the tubing would be affected by the residual stress system in the material.

The residual stresses in the Integron were estimated by machining slices longitudinally from lengths of the tubing and measuring the resulting curvature. This method was used to investigate the stress levels remaining after heating the Integron tubing for 24 hours at temperatures up to 400° C., and also after heating for various periods of time up to 190 hours at 300° C. This data indicated the stress levels that would be present after the tubing had been in service.

The distribution of the longitudinal and tangential stresses was investigated in the Integron tubing in the " as-finned " condition. Longitudinal slices were reduced in thickness by machining and electrolytically dissolving material from one face, and the curvature corresponding to each condition was determined. The longitudinal stress distribution was calculated by a method similar to that used by both Sachs and Espey[1] and Richards[2], making assumptions appropriate to a thin walled tube and ignoring the effect of the finning. In the tangential direction the stresses relieved by slitting a ring of the Integron longitudinally, and also by subsequently removing the fins, were estimated.

Fatigue Behaviour

The turbulence of the gas may cause a certain amount of vibration in the tubing of some heat exchanger structures ; it was therefore considered important to establish that the fatigue properties of the Integron were satisfactory in both the " as-finned " and heat treated conditions. No rig was available for quantitative fatigue tests on whole sections of Integron tubing at this time, but some work on whole sections, carried out by Imperial

Chemical Industries, Ltd., Metals Division, Research Department, had shown that in rotating bending, the fatigue crack would start from the grooves between the fins on the outside surface of the tubing. In consequence it was considered satisfactory to carry out tests on sectors cut longitudinally from the tube wall. The sectors were constricted to ensure that when vibrated as a free cantilever, they fractured in a zone comprising four of the grooves between the fins. The cutting of the sectors relieved compressive residual stresses in the fracture zone, and the "as-finned" Integron tubing was therefore tested in a less favourable condition than might obtain in service.

The tests were carried out at room temperature on Integron tubing in the normalised, sub-critically annealed (625° C.) and "as-finned" conditions. Before being tested the sectors were heated for 24 hours at 300° C. in air to simulate some of the effects of the service temperature conditions. A staircase testing procedure[3] was used to provide data for the estimation of the three fatigue limits. A total of 39 specimens were tested to "cracking," or to "no cracking" after an endurance of 2×10^7 cycles, the criterion of cracking being a 3% drop in the resonant frequency of the specimen.

The vibration rig, which is shown in Fig. 2, had been developed by D. A. Davis and F. Moran, at the Central Electricity Research Laboratories for work on turbine blade vibration. The specimen was clamped between hardened steel blocks, and excited in resonant transverse vibration in the vertical plane by an electromagnetic vibrator. The resonant frequency of the system was normally between 240 and 250 c/s. The electronic system of amplitude control used a stationary capacitance probe which can be seen immediately below the end of the specimen in Fig. 2. Throughout the tests, under non-breaking conditions, the amplitude was held constant to within $\pm$ 2%. The relative amplitude of vibration of the specimen was indicated by an optical lever. A concave mirror attached firmly to the end face of the specimen reflected a spot of light on to a scale. The scale and lamp of the optical lever are not visible in Fig. 2. The frequency of vibration of a specimen fell under conditions of crack propagation, because the system was self-actuating and normally vibrated in resonance, and the control system was designed to stop the test after any desired drop in frequency.

A calibration was made to relate the deflection of the optical lever to the longitudinal stress (ignoring the effect of the finning) in the zone from which fracture started. A specimen was mounted in the rig and uniformly loaded along its length under static conditions. This type of loading gave a deflection curve similar to that given by the inertia loading during resonant vibration, and the stress level at the required position was readily estimated from the measured data.

Welding

In current nuclear power station designs, the finned heat exchanger tubing is flash butt-welded or oxy-acetylene welded to unfinned tubing, and the welds are usually normalised by heating with a gas flame. Zones of metal, cold worked to a critical degree of strain, could be subject to grain growth when heated to 700°–750° C. and held in that temperature range for a sufficient length of time.

Some lengths of Integron were oxy-acetylene butt-welded together, and sectioned for microexamination and tensile tests, to determine whether zones of this kind containing coarse grains were likely to be developed in the Integron by the welding procedures.

TABLE I.—PRINCIPAL DIMENSIONS OF INTEGRON HEAT EXCHANGER TUBING FOR BRADWELL AND BERKELEY POWER STATIONS.

Dimension	Integron for Bradwell	Integron for Berkeley
	in.	in.
Bore diameter	1·50	0·88
Wall thickness	0·25	0·18
Fin spacing	0·19	0·14
Fin thickness..	0·06	0·055
Fin height	0·3	0·3

Experimental Results

Dimensions, Variations and General Features

Visual examination of the Integron mild steel tubes indicated that both types were sound, reasonably straight, and free from defects likely to affect their behaviour in service.

Typical dimensions of the two types of Integron are given in Table I. Numerous measurements of wall thickness were made on the tubes of Integron for Bradwell that were cut to obtain fatigue test specimens. The results varied between 0·233 in. and 0·275 in., and the circumferential variation did not exceed 0·03 in. for any one transverse section of the tube. It was apparent that variations in the wall thickness of the original tubing were not greatly exaggerated by the finning process, and that the finned surfaces of the Integron tubing were rolled true, most of the variations in wall thickness being apparent as irregularities of the bore.

Longitudinal bore defects in the seamless Integron tubing were mostly less than 0·002 in. in depth. The bore diameter may be slightly increased in the Integron rolling process, but there was no significant extension of irregularities on the bore of the tubes examined. A ridge or flash of metal running circumferentially around the tube was present on one side of successive fins of one of the tubes (Fig. 3). This was probably caused by a damaged tool.

The Berkeley Integron, rolled from ERW tube, contained a small continuous lap or crack at the tips of the fins, Fig. 4. The bore marks left by the tool used to remove the internal weld flash, during the manufacture of the tube, were not significantly altered by the finning process.

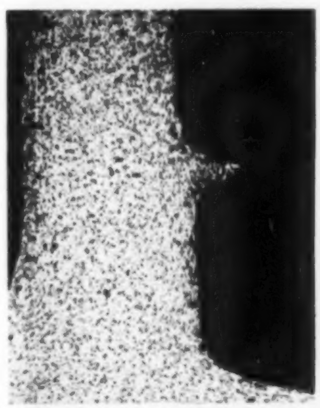


Fig. 3.—Section showing circumferential flash on finning of a length of Bradwell size Integron. ×15

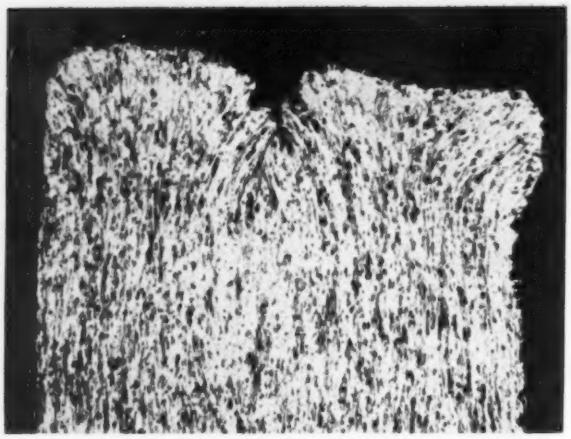


Fig. 4.—Fin tip profile in Integron tubing for Berkeley. $\times 50$

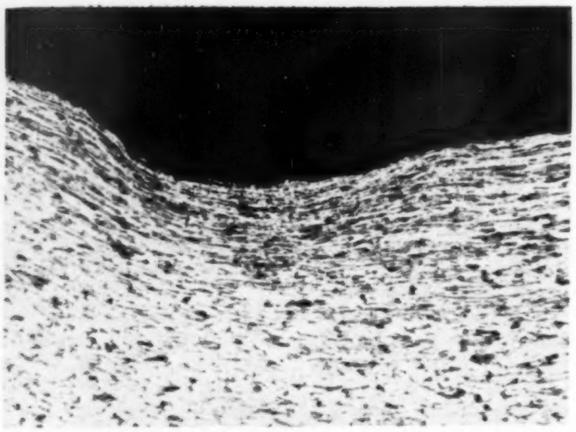


Fig. 5.—Longitudinal section of Integron tubing showing roll mark defect at the base of one of the grooves. $\times 50$

The surface of the metal in the grooves between the fins was smooth and free from tears, but there were numerous roll marks, which had the form of shallow depressions. A section through one of the sharpest of these is shown in Fig. 5. The profile radii on either side of the fin were unequal to a varying extent on different tubes, but they were quite satisfactory in all the tubes examined. Typical profiles are shown in Figs. 6a and 6b.

Microstructures

In both sizes of Integron tubing the structure showed definite indications of cold work in the fins and to a distance of about 0·02 in. below the grooves (see Figs. 3, 4 and 5). The microstructure in the remainder of the tube wall showed no evidence of cold working, and the grain size varied from ASTM 6–7 in the Bradwell tubing to ASTM 7–8 in the Berkeley tubing.

Sub-critical annealing at 625° C. caused the ferrite in the heavily cold worked regions to recrystallise with a grain size of ASTM 8 or less, as shown in Fig. 7, but the grain size in the body of the tube wall was not affected. Sub-critical annealing also spheroidised the pearlite. The time of annealing was not critical within the limits 15–100 min., and the same structural effects were produced with an annealing treatment at 650° C. for 1 hour. Material heat treated at 675° and 700° C., however, showed a thin band of coarser grains (up to ASTM 5) extending below the grooves and fins.

Normalising in a protective container gave a uniform structure with a grain size varying between ASTM 7 and 8. Heat treatments for 24 hours at temperatures up to 450° C. did not cause any apparent changes in the microstructure.

Hardness Determinations

The results of hardness surveys made on the Bradwell Integron tubing in various conditions of heat treatment are shown in Fig. 8. The fall in hardness became significant for heat treatments over 400° C., and at 625° C. the heavily worked regions were very largely softened. Normalising was necessary completely to remove hardening in the material. This treatment reduced the hardness in the tube wall by about 20 units (V.P.N.). A carefully controlled test showed that heating the "as-finned" material at 200° C. produced a hardness increase of 10–15 V.P.N. over the entire tube wall section.

Micro-hardness impressions, made to within 0·002 in.

(a)

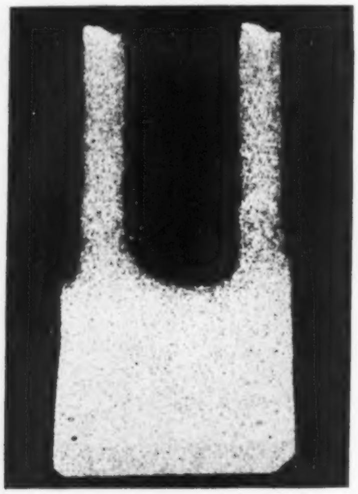
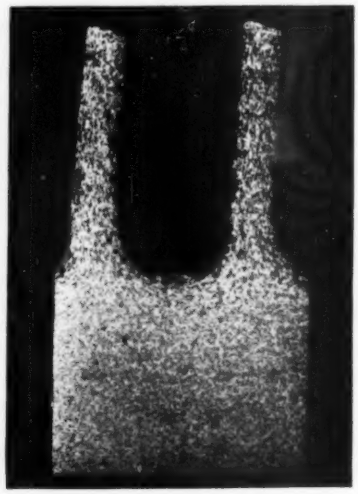


Fig. 6.—Longitudinal sections of Integron for Bradwell showing (a) tube with unequal fin root radii, and (b) tube with fin root radii nearly equal. $\times 5$



(b)

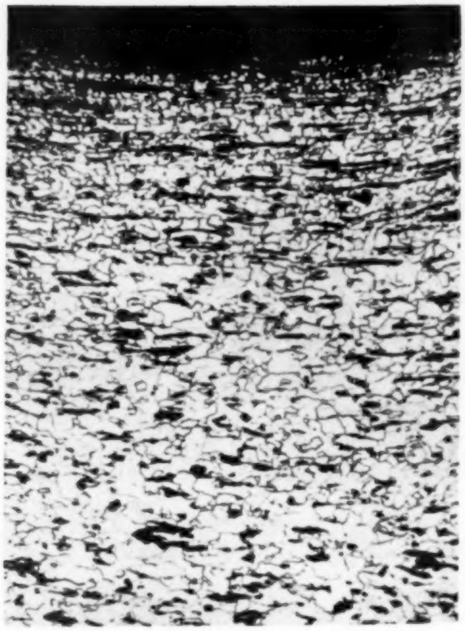


Fig. 7.—Longitudinal section of sub-critically annealed (625° C.) Integron showing the microstructure below a groove. **×100**

of the groove surface, showed a continuous increase of hardness towards the surface and a probable maximum in the ferrite of 250 kg./sq. mm. (100 g. load). Micro-hardness values were similar to the macro-hardness values in regions where the surveys overlapped.

Tensile Tests

The results of the room temperature tensile tests on testpieces machined longitudinally from the tube wall of the Bradwell Integron tubing in the "as-finned" and heat treated conditions are shown in Table II. The results, which are the average of three tests, indicate that the material in the centre of the tube wall is quite ductile in all three conditions.

The results of similar tests carried out at temperatures up to 450° C., thus covering the service temperature range, are shown in Fig. 9. The tensile properties of material in the "as-finned" condition were compared with those of the material in the normalised condition. The effect of the strain age-hardening on the tensile strength was not very great, the maximum effect being at 300° C. There was a large difference in the limits of proportionality at 300° C.

Residual Stresses

High longitudinal compressive stresses were indicated along the groove bases. Tensile stresses were present in the fins and along the tube bore. The estimated

TABLE II.—TENSILE PROPERTIES OF SPECIMENS MACHINED FROM THE TUBE WALL OF INTEGRON HEAT EXCHANGER TUBING

Condition	Tensile Strength (tons/sq. in.)	Yield Point (tons/sq. in.)	Elongation (% on $4\sqrt{A}$)
"As-finned"	26·1	none (L.O.P.$\approx$23)	37
Sub-critically annealed (625° C.)	26·1	18·4	38
Normalised	26·4	17·2	43

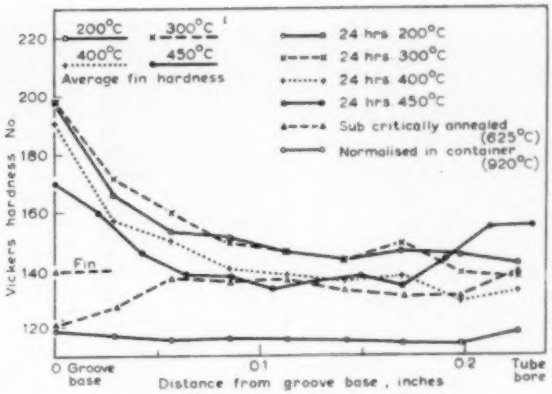


Fig. 8.—Relief of cold work hardening in Integron by heat treatment.

longitudinal stress distribution in the tube wall is shown in Fig. 10: the effect of the finning was ignored. The principal feature is the distinct knee in the curve at about 0·04 in. from the bore. It suggests that the tensile stresses near the bore had been relieved by plastic flow subsequent to the rolling. Thermal and mechanical strains imparted as the Integron tubing left the mill might have been responsible.

In the tangential direction cutting relieved high stresses only in the finning. The highest stresses were tensile in nature, and in one of the two tubes examined they were three times as great as in the other tube, probably approaching the elastic limit of the fin material (i.e., about 40 tons/sq. in.).

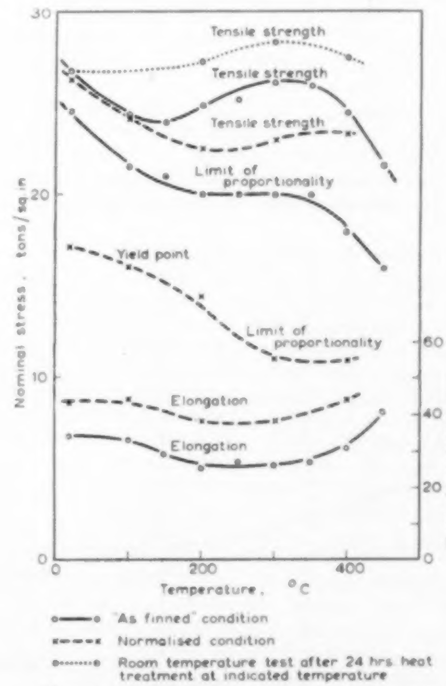


Fig. 9.—The effect of high temperatures on the tensile properties of test pieces machined from the tube wall of Integron.

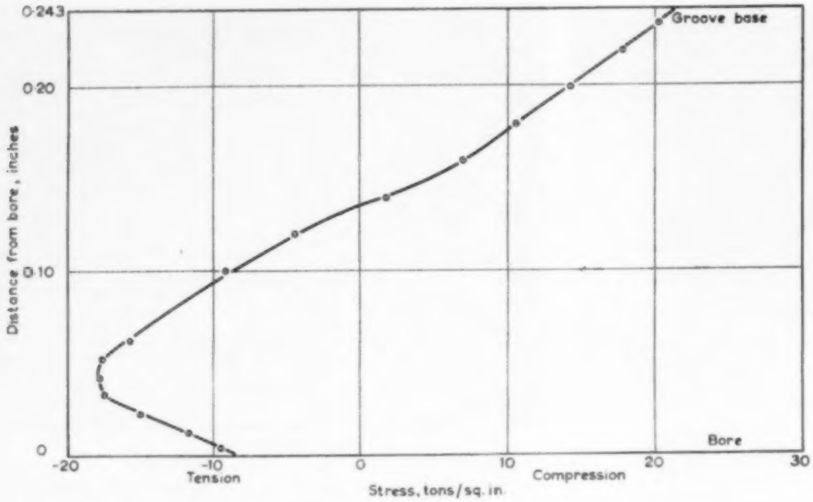


Fig. 10.—Distribution of average longitudinal residual stress in wall of Integron tube.

The relaxation tests showed that after a few days heating at 300° C. the stresses were reduced overall to about two-thirds of their original value, most of the reduction occurring in the first 24 hours. After 24 hours at 400° C. the stresses were reduced overall to less than half their original value. These results are shown in Figs. 11 and 12.

Fatigue Tests

The results of the cantilever bending fatigue tests are shown in Table III. The relative fatigue limits assume the fatigue limit of the normalised sectors to be 100.

TABLE III.—THE EFFECT OF HEAT TREATMENT ON THE FATIGUE STRENGTH OF INTEGRON SECTORS.

Condition	Fatigue Limit (tons/sq. in.)	Relative Fatigue Limits
Heat treated 300° C. only	12·2	125
Sub-critically annealed (625° C.)	10·6	107
Normalised	9·8	100

An analysis showed that the difference between the results for the two heat treated conditions was significant. The roll marks in the grooves did not appear to affect the behaviour significantly.

Welding

The welded Integron tubing was sectioned, and micro-examination showed that no increase in the grain size had occurred in the heat-affected zone, beyond that previously existing in the centre of the tube wall, disregarding the coarse Widmanstätten structure immediately adjacent to the weld metal. The structure of the region heated to a temperature just below the transition range was similar to that of Integron sub-critically annealed at 625° C. The ferrite in the heavily cold worked metal near the base of the fins had recrystallised with a fine grain size, as seen on the right hand side of

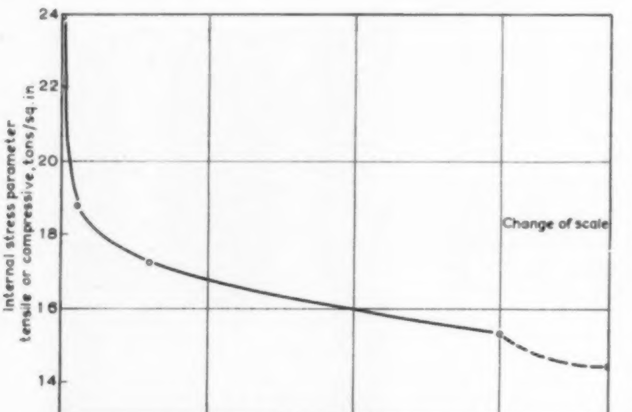


Fig. 11.—Relaxation of longitudinal internal stress at 300° C. in Integron.

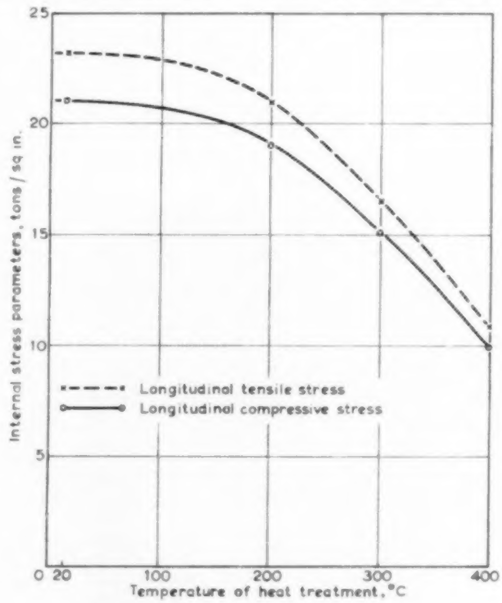


Fig. 12.—Relief of internal stress by 24 hour heat treatments.

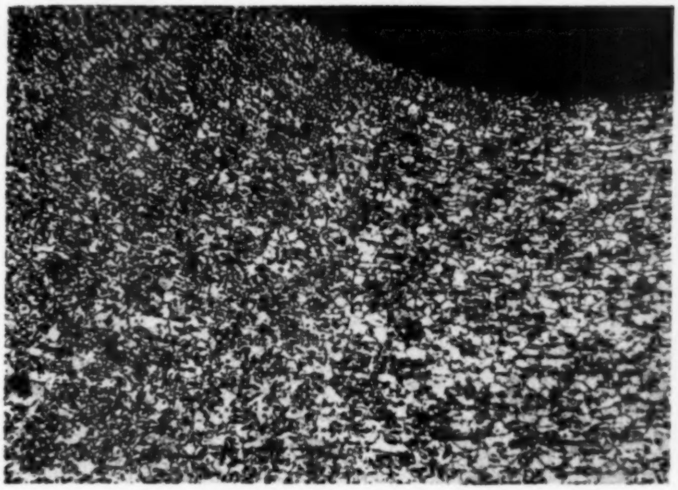


Fig. 13.—Part of weld heat-affected zone in Integron showing, on the left, material heated into the critical range, and on the right material heated to just below the critical range. ×50

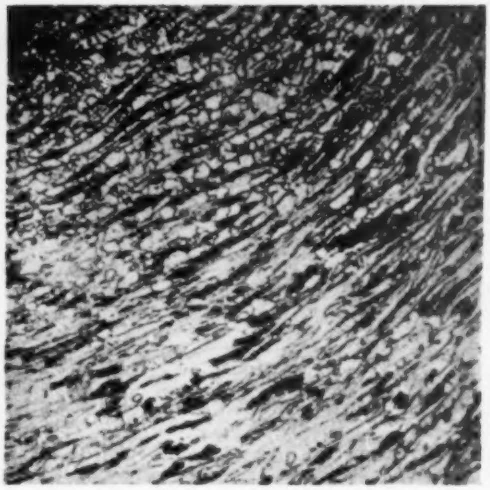


Fig. 14.—Outer part of weld heat-affected zone in Integron showing recrystallised grains in heavily cold worked metal near a groove. ×200

Fig. 13. On the left hand side of Fig. 13 the metal had been heated into the phase transition range and was in consequence grain refined.

Tensile tests were carried out on slices containing a weld, which had been cut longitudinally from the Integron. Fracture occurred in one or other of the zones heated to just below the transition range (i.e., at 1–1¼ in. from the weld), but there was a large amount of plastic deformation in the regions nearer the weld. It was concluded, therefore, that no highly localised zone of weakness had been produced anywhere in the heat-affected zone. Fig. 14 shows the outer part of the heat-affected zone in which only the most heavily worked regions have recrystallised. This area was approximately 2 in. from the weld.

Conclusions

The Integron tubes were smooth, straight and regular in form, and they contained no defects which would appreciably affect their behaviour in service. Longitudinal bore defects present in the original tubes had not been significantly extended during finning, and wall thickness variations in the Bradwell Integron probably followed variations in the thickness of the seamless tube before finning.

The rolling process severely cold worked the fins and a layer of material at least 0·01 in. thick at the base of the grooves, resulting in hardnesses in excess of 200 V.P.N. Most of the remainder of the tube wall had been slightly work hardened, the hardness being about 20 units (V.P.N.) higher than normalised material, but the microstructure showed no indication of cold work in this region. Tensile tests on specimens taken from the centre of the tube wall gave stress/strain curves with no yield point, the limit of proportionality being only 3 tons/sq. in. below the tensile strength. The finning process increases the length of the tubing by about 5%, and should not increase or decrease the bore diameter by more than a similar amount. The effects on the material in the tube wall were consistent with this amount of strain.

The strain age-hardening effects were greatest at 300° C., but they were of small magnitude compared with the effects of work hardening, as would be expected in a mild steel fully killed with aluminium.

When residual stresses are present in a metal component the value of the externally applied stress needed to cause plastic flow and hence permanent distortion, is altered. Permanent distortion will be produced when, at any point within the material, the residual stress plus the applied stress exceeds the elastic limit. The residual stresses in the Integron tubing "as-finned," were compressive along the outer part of the tube wall and tensile along the bore. The form of the longitudinal stress distribution indicated that some degree of stress relief had occurred by plastic flow in the material adjacent to the bore, and in consequence the tubing must, in the condition examined, have had some resistance to permanent distortion when externally stressed.

The results of the relaxation and tensile tests indicated that when the tubing is heated to service temperatures it should become more resistant to permanent distortion. At 300° C., for example, the residual stresses are lowered by a greater amount than is the elastic limit of the material in the wall of the tube. Appreciable residual stresses remained in the Integron tubing at service temperatures, however, and these would increase the resistance of the grooved surface to fatigue cracking.

The fatigue tests showed that the grooved outer surface had a satisfactory resistance to fatigue failure in both the heat treated and "as-finned" conditions. The tubing in the "as-finned" condition had a fatigue limit at least 25% higher than the fatigue limit of the tubing in the weakest heat treated condition (normalised in cast iron borings). Additional fatigue tests at the Central Electricity Research Laboratories on complete Integron tubes, vibrated in resonance in the free-free mode, have confirmed both the path of failure and the value of the fatigue limit for "as-finned" Integron.

There was no increase in the maximum grain size in Integron joined by oxy-acetylene gas butt-welding, out-

side zones heated through the critical range, and tensile tests did not reveal any serious local weakening. The welding procedures used in fabricating the Berkeley and Bradwell heat exchangers should produce similar results in the heat-affected zones.

To summarise, all the investigations carried out showed that mild steel Integron tubing should be satisfactory for service without heat treating under the conditions proposed for the Bradwell and Berkeley heat exchangers.

Acknowledgments

This work was instigated at the request of the Technology Branch of the C.E.G.B. Research and Development Department. The authors would like to record their thanks to their colleagues Mr. D. A. Davis and Mr. F. Moran, who developed the vibration rig used for the fatigue tests, and whose collaboration was essential in carrying out this part of the work, and to members of the Research Department of I.C.I. Metals Division for their assistance in the early stages of the investigations. The authors would also like to thank the Director of the Central Electricity Research Laboratories for permission to publish this paper.

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Davy-United Steel Processes Division

Davy and United Engineering Co., Ltd., announce that they have formed a Steel Processes Division to absorb the personnel and carry on the work previously undertaken by Davy British Oxygen, Ltd. Davy British Oxygen was established over three years ago jointly by Davy-United and British Oxygen to investigate the various kinds of pneumatic steelmaking processes and to carry out the basic engineering studies necessary for the design of complete oxygen steelmaking plants. This arrangement brought together the plant designing resources of Davy-United and the techniques in the use of oxygen which the British Oxygen Company has developed.

The rapid expansion of oxygen steelmaking throughout the world has necessitated an expansion of activity by the joint company, and the two companies have agreed that the execution of prospective contracts for oxygen steel melting shops should be handled by Davy-United in view of their experience of large-scale contracts involving heavy steelworks plant. Accordingly the Steel Processes Division has been formed, although Davy British Oxygen, Ltd., will remain in existence and act as the focal point for future collaboration in this field. Davy-United's Steel Processes Division is at Suffolk House, Suffolk Road, Sheffield 2, and is staffed and equipped to provide metallurgical, technical and economic surveys against any projected steelmaking development; design services for oxy-steel equipment; and the engineering and supply of oxygen steelmaking plant up to complete melting shops.

Albert Mann Engineering

As a result of the conclusion of a licensing agreement with Loma Machine Manufacturing Co., Inc., and its subsidiary, Lobeck Casting Processes, Inc., of New York City, and of the taking over of the whole of the share capital of Roll Race Conveyors, Ltd., the Albert Mann Engineering Co., Ltd., of Basildon, Essex, can now offer to the British and West European metal industries their modern designs of rolling mills and ancillary equipment; slitters; roller levellers, etc.; the range of continuous non-ferrous strip casting and rolling lines; the range of high production processing machinery of Loma and Lobeck design including billet and slab casting equipment; circular saw machines; continuous rod and tube drawing, straightening and cut-off lines; stretcher levellers; air and hydraulic tube testers and all related mechanical handling equipment, including the Roll Race system, together with electronic design and manufacturing facilities to drive, control and integrate complete schemes.

Nickel-Aluminium Bronze Propellers

From M.T.B.'s to the latest in super tankers, that is the story of the development of nickel-aluminium bronze propellers. In 1942, after a series of failures of high tensile brass propellers, the use of nickel-aluminium bronze was recommended to the Admiralty, tested, and immediately adopted as the standard material for motor torpedo boat propellers. Last August, a five-bladed propeller cast at the works of Lips N.V., Drunen, in Holland, had a net weight of 39 tons and a cast weight of about 50 tons. This is the largest propeller ever cast in nickel-aluminium bronze, with a diameter of 7·5 m., and it is to be used on a 72,600-ton super tanker being built at Dunkirk by Ateliers et Chantiers de France for the Tidewater Oil Company. The super-tanker will be equipped with only one propeller, which means that great care had to be taken in its manufacture and indicates the confidence the builders and the tanker's owners have in the performance of the propeller itself.

There are several reasons why this type of propeller is popular and why increased uses for nickel-aluminium bronze can be predicted. It is a lighter, yet stronger material, and has a high resistance to corrosion fatigue in sea water. The thickness of the blades can be reduced without losing strength, and this means less weight and more efficiency. In many cases a saving in weight of 20% can be effected over the more conventional high-tensile brass propeller.

The reduced weight means less wear on shaft bearings, less power required and, of course, that a 2-ton saving in propeller weight enables a ship to carry 2 tons extra cargo. In the case of a ship burning 50 to 60 tons of fuel a day, increased propeller efficiency could mean a financial saving of at least £7 a day.

Finally, the very important matter of maintenance costs should be considered Nickel-aluminium bronze has a greater resistance to corrosion and erosion than ordinary high tensile brass, and since corrosion is metal's worst enemy in marine applications this factor is very important. The use of these propellers on the U.S. Navy's arctic exploration voyages is an example of nickel-aluminium bronze's great strength and erosion resistance.

Some Factors Affecting the Intergranular Brittleness of Internally Oxidised Alloys

By R. H. Seebohm and J. W. Martin.

(*Department of Metallurgy, University of Oxford*)

Tensile tests have been carried out on copper dispersion-hardened by internal oxidation. The results presented and discussed show that the intergranular brittleness (associated with intense grain boundary precipitates) was eliminated when specimens were prepared by recrystallisation of internally oxidised single crystals. It was also demonstrated that the extent of solute segregation to the grain boundaries could be reduced (and hence the intensity of precipitation there on internal oxidation) by introducing preferred orientation into the specimen.

THE work described in this paper forms part of an investigation into the relationship between the microstructure and the mechanical properties of alloys containing a fine dispersion of an oxide phase. The technique of internal oxidation has been employed to produce a dispersed oxide phase in copper. An alloy suitable for this treatment consists of a dilute solid solution of a base metal in a more noble metal ; the alloy is heated under oxidising conditions, so that oxygen diffuses into the alloy and produces a dispersion of the oxide of the base metal in a matrix of the noble metal.

Chaston[1] and Meijering and Druyvesteyn,[2] working independently, were the first to report hardening effects caused by internal oxidation. These results have prompted further studies of the effects of internal oxidation on hardness,[3-7] tensile,[8-10] creep[11-12] and fatigue[13] properties of a number of alloys. Plastic deformation usually leads to intergranular fracture of these materials (see, for example, references 3 and 8) due to oxide being concentrated in the grain boundaries as well as dispersed within each crystal.

Two series of experiments are described below : the first was designed to reduce the intergranular brittleness of these materials by eliminating the oxide precipitate in the boundaries. The second series is an attempt to control the extent of the segregation of the solute to the grain boundaries, and thus to reduce the intensity of precipitation there on internal oxidation.

Experimental

I—The Effect of Recrystallisation on the Tensile Properties of an Internally Oxidised Copper Silicon Alloy

Gregory and Smith[8] have reviewed the factors affecting the formation of the dispersed phase, and they emphasise that a large difference between the free energy of formation of the oxide of the solute and the oxide of the solvent favours the formation of small particles. Thus, the particles formed on the oxidation of silver-aluminium alloys are far smaller than those obtained in silver-silicon alloys, which, in turn, are smaller than those formed in copper-silicon alloys under the same conditions.

In the present work, by selecting a copper–0·3% silicon alloy, the coarser oxide dispersion produced has enabled successful recrystallisation experiments to be carried out on internally oxidised material, and some tensile properties of the resultant structure have been determined. Some observations of the recrystallisation kinetics have been published elsewhere.[14]

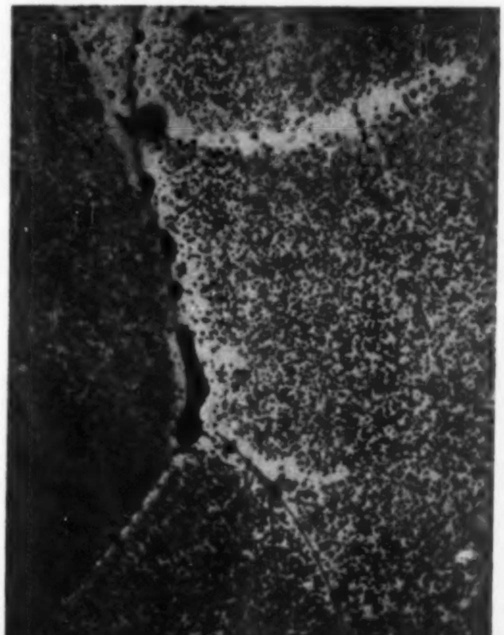


Fig. 1.—Micrograph of copper—0·3% silicon wire tensile specimen internally oxidised at 950° C. Etched ferric chloride ×600

Tensile tests have been carried out using a Hounsfield "Tensometer," and wire specimens 0·036 in. in diameter, and of 2 in. gauge length, held in special friction grips.[8] Wires of three types of structure have been tested :

(*a*) Copper–0·3% silicon wire annealed at 950° C.

(*b*) Wire of the above composition and grain size internally oxidised at 950° C. This was carried out by packing the specimens in a copper powder/cuprous oxide mixture, which provided a partial pressure of oxygen equal to the dissociation pressure of cuprous oxide at the temperature employed. Thus, no

Fig. 2.—Electron micrograph of carbon extraction replica (Au-Pd shadowed) of copper—0·3% silicon wire tensile specimen in the form of a single crystal internally oxidised at 950° C. and recrystallised at 800° C. ×1,400

external scale was formed on the wires during the oxidation treatment. The microstructure of a typical specimen appears in Fig. 1.

(c) Single crystal specimens of copper–0·3% silicon grown in the form of $\frac{3}{16}$ in. diameter rods, and internally oxidised at 950° C., then drawn to 20 s.w.g. wire (0·036 in. diameter), and annealed *in vacuo* for 144 hours at 800° C.

Fig. 2 shows the microstructure obtained by the last process. There is no evidence of concentration of oxide in the grain boundaries, nor any trace of particle denudation in the neighbourhood of the boundary. Both of these effects characterise the microstructure of oxidised polycrystalline material (Fig. 1).

Fig. 3 shows typical load-elongation curves obtained with these three types of specimen. *A* is a curve obtained with a copper–0·3% silicon wire annealed at 950° C., and *B* that for a wire of this grain size internally oxidised at 950° C. An increased rate of work-hardening is seen to result from the internal oxidation, but the ductility is decreased and the fracture is intergranular. Curve *C* is for a recrystallised single crystal: while retaining the increased rate of work-hardening compared with the annealed material, the ductility is greater than that obtained with the annealed specimen. The fracture was fibrous in texture and followed a transcrystalline path.

II — The Effect of Texture on the Tensile Properties of an Internally Oxidised Copper-Beryllium Alloy.

The production of tensile specimens from rolled alloy strip which possessed, after internal oxidation, an anomalously higher ductility than previously reported for this type of material, has prompted a study of the effect of the shaping operation upon the ductility of oxidised specimens. An alloy of copper with 0·21% beryllium was selected, and the tensile behaviour of oxidised specimens machined from rolled strip has been compared with that of similar specimens machined from extruded rod. (A number of wire specimens were also prepared and tested for comparison purposes).

Materials

Two sets of flat strip specimens were prepared: one set was machined from 0·0725 in. × $\frac{5}{16}$ in. rolled strip, and the other from $\frac{5}{16}$ in. diameter extruded rod, milled into 0·0725 in. × $\frac{5}{16}$ in. strip.

Heat Treatment

All specimens were given a preliminary anneal *in vacuo* for 1 hour at 1,000° C., in order to stabilise the grain size. This was followed by the internal oxidation treatment, which consisted of holding at 1,000° C. in a furnace, with air being drawn through for 1 hour, the atmosphere being changed to argon thereafter, and the heat treatment continued for a further 8 hours. Thus the copper oxide film produced initially subsequently became the source of oxygen for the complete internal oxidation of the specimen during the argon treatment.

Specimens

After internal oxidation, a waist of width 0·22 in. was milled in each specimen, with a fillet radius $\frac{1}{8}$ in., and a 1 in. gauge length was scribed on each specimen.

Structure

An electron micrograph of a carbon extraction replica from this material appears in Fig. 4. The grain-size of all specimens tested was in the range 0·05–0·08 cm. diameter.

Mechanical Properties

After measuring the Vickers diamond pyramid hardness of each surface, the specimens were again tested on a "Tensometer," the percentage elongation being measured by means of vernier callipers on the 1 in. scribed marks: the fracture always took place within the marked gauge length. The results of these tests are set out in Table I. A similar effect has been observed in a series of rolled and of extruded alloys of copper–0·25% aluminium, after internal oxidation.

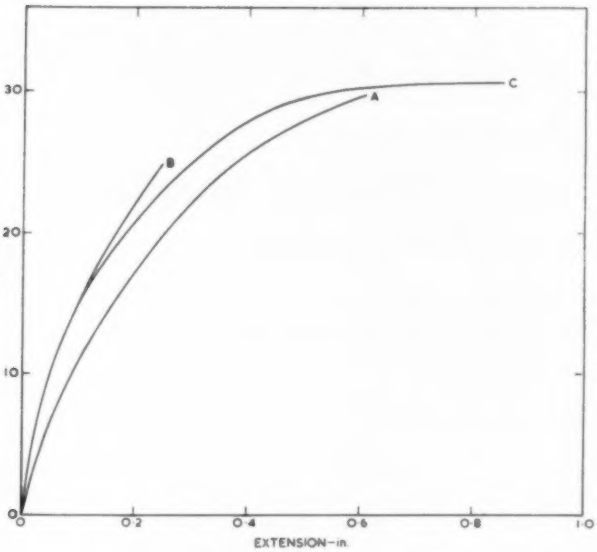
A number of wire specimens of the copper-beryllium alloy, with diameters ranging from 0·0825 in. to 0·0406 in., were also prepared (as described for the copper-silicon wires above) and tested in tension. In each case extreme brittleness was encountered, the tensile results, in fact, having little significance because of this: the elongations were in the range 0–2%.

Although X-ray cylindrical and flat-film photographs have been taken of Debye-Scherrer rings from rolled and from extruded and drawn specimens, the grain sizes were all too large for satisfactory patterns, and no pronounced textures could be detected.

Discussion

Recrystallisation Experiments

Curves *A* and *B* in Fig. 3 are in agreement with the results of other workers, e.g. Gregory and Smith[8] on silver alloys, while curve *C* indicates that the inter-



A Annealed 950° C.
B Internally oxidised at 950° C.
C Single crystal internally oxidised at 950° C. and recrystallised at 800° C.

Fig. 3.—Load v. elongation curves of copper—0·3% silicon specimens of 0·036 in. diameter: gauge length 2 in.

Fig. 4.—Electron micrograph of carbon extraction replica (Au-Pd shadowed) of copper—0·21% beryllium internally oxidised at 1,000° C. ×10,000

granular brittleness in specimen *B* must be associated with a grain boundary concentration of oxide. This will be in the form of particles, or as a continuous precipitate, and is presumably due to a segregation of solute atoms (silicon) to the grain boundaries of the copper prior to oxidation treatment.

Texture Experiments

A number of papers have recently discussed the relationship between grain orientation and precipitation in grain boundaries, particularly in age-hardening systems.[15, 16] Where misfit is small, precipitation becomes less dense. Forsyth, Metcalfe, King, and Chalmers[17] show a photomicrograph of a circular grain included within another grain of aged copper–1·8% beryllium, such that precipitation varies in intensity round the circumference of the included grain from vanishingly small to a thickness of several microns.

It is suggested that the grain boundary precipitates produced on internal oxidation vary in a similar way, and that intergranular failure will take place increasingly readily as the grain boundary precipitate becomes more intense. Thus the higher ductility obtained with the rolled specimens as compared with that obtained with extruded or drawn material, may be explained if one assumes that they contain a higher proportion of low-angle boundaries. Even after recrystallisation, any rolling texture must increase the proportion of low-angle boundaries—indeed the ultimate preferred orientation is of course a single crystal, with its high ductility and evenly distributed precipitate.

TABLE I.—RESULTS OF TENSILE TESTS ON EXTRUDED ROD AND ROLLED STRIP IN COPPER–0·21% BERYLLIUM ALLOY.

	Elongation (%)	Vickers Diamond Pyramid Hardness	Ultimate Tensile Stress (tons/sq. in.)
Rod ..	4·1	120	14·45
	3·2	115	12·9
Strip ..	10·5	124	15·55
	7·7	115	13·8

Previous workers in this field have worked with specimens made from rods or wires (see, for example, references 3 and 8), and although they may have a fibre texture—i.e. a tendency for a certain crystallographic direction to lie along the specimen axis, they will presumably have a normal distribution of high-angle boundaries, and extremely low values are reported for the ductilities of these materials after oxidation. The effect of a preferred orientation (i.e. of crystallographic planes *and* axes), and the subsequent production of low-angle boundaries has been investigated by a few workers studying intergranular attack,[18, 19] and a slight effect attributable to this cause has been observed in that field also.

It may be claimed that the experiments described here might illustrate a general method of controlling ductility in precipitation-hardening systems. In cases, for example, where creep ductility is impaired by the presence of intergranular precipitates, it is conceivable that a prior forging operation might cause the development of sufficient preferred orientation for an appreciable improvement in ductility to be observed. Further research on these lines would be interesting.

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Oil Circulation in Porous Sintered Metals: Note on Rate of Absorption

By Dr. J. Heuberger

Aktiebolaget Glissa, Indstrumentvägen 21, Stockholm Hagersten.

POROUS self-lubricating sliding bearings are one of the oldest industrial products of powder metallurgy in modern times. Nevertheless, our knowledge of the function of the pores in this material is still very incomplete. As a rule, the inter-connected pore system (the pore volume) is considered only as a static storeroom for the bearing oil. The oil flow taking place in the pore system, however, has seldom been the subject of attention, and ideas regarding the prevailing quantitative relationships appear to have been vague. This supposition is supported by the following example.

In a vertically mounted self-lubricating sliding bearing, there is a danger that oil from the bearing face may run along the shaft and be lost. In order to return this oil into circulation in the bearing, a construction of the type shown in Fig. 1 may be used. This presupposes that, in consequence of capillary forces, the bushing absorbs oil so quickly that no oil impoverishment of the bearing takes place. Since there was reason to suppose that this condition is not always fulfilled, some exploratory experiments were carried out to determine the rate of rise of oil in a porous sintered steel bushing.

A sintered steel bushing of dimensions 34/40 × 77 mm. was marked on the outside with a scale showing 0 to 77 mm. This bushing, in the unimpregnated condition, was placed in a dish containing oil, and the height to which the oil rose was read directly on the scale. The bushing used had a porosity of 33 volume %, and the pore size varied from 0 to 0·2 mm. The oil used in these experiments was of a viscosity SAE 50. Experiments were carried out at + 6°, + 18°, and + 75° C., and the results are plotted in Fig. 2. It will be seen that the rate of rise of the oil in the bushing is very strongly temperature dependent, and, even at the highest temperature used, is by no means as rapid as would be generally supposed.

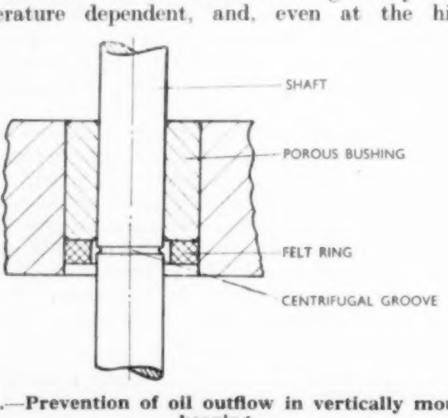


Fig. 1.—Prevention of oil outflow in vertically mounted bearing.

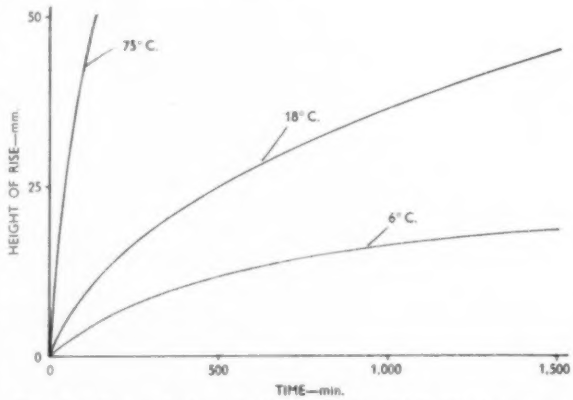


Fig. 2.—Rate of rise of oil (viscosity SAE 50) in porous sintered steel bushing.

The theoretical evaluation of these curves is attended with certain difficulties since, among other things, the pore channels in a sintered steel bushing cannot be defined by means of simple physical quantities: owing to variations of diameter, both laminar and turbulent flow take place. With rising temperature, the diameter of the pore channels increases, the viscosity of the oil decreases, and with it the specific gravity of the oil, and, consequently, the counter-pressure of the oil column. A more complete investigation of these relationships, under varying conditions of sintered material, porosity, pore distribution, and oil, is projected.

PLUTONIUM FURNACE ORDER

THE GENERAL ELECTRIC CO., LTD., has received a further contract from the United Kingdom Atomic Energy Authority, Risley, for electric furnaces for use in the production of plutonium metal. This latest order covers reduction and fluorination furnaces. The fluorination furnace is rated at 10 kW., and is of the horizontal front-loading type, complete with muffles, the inner surface of the muffles and doors being lined with platinum. A forced cooling system is incorporated to enable the charge to be cooled within a stipulated time. The furnace for the reduction process is rated at 50 kW. It is of a bell-type design and has a forced cooling system similar to the fluorination furnace. Electrical equipment being supplied includes regulating transformers, temperature control gear, and the necessary switchgear.

The Application of X-Ray Scanning Microanalysis to Some Metallurgical Problems

By D. A. Melford, M.A., Ph.D., and P. Duncumb, M.A., Ph.D.

Tube Investments Research Laboratories, Hinxton Hall, Cambridge.

The value of the scanning technique in the study by X-ray microanalysis of the distribution within a metal of a particular constituent or impurity is illustrated by reference to applications concerned with inclusion analysis ; the effect on hot-shortness of surface segregation in mild steel ; the segregation of lead in free-cutting steels ; the analysis of individual phases in a three-phase alloy ; and analysis at fracture surfaces. The authors conclude with a consideration of the problems involved in the extension of microanalysis to elements of atomic number less than 12.

THE technique of electron probe microanalysis initiated by Castaing[1] has been applied with great success to many metallurgical problems in the last three or four years, for example by Philibert and his co-workers.[2] It consists of using a focused electron beam, normally accelerated to 20–40 kV., to excite X-rays from a region in the surface of the specimen approximately 1 micron in diameter and rather less than this in depth. These X-rays are then subjected to spectral analysis so that in principle it is possible to obtain the complete chemical composition of this region *in situ*.

For the metallurgist this development has opened up new possibilities for studying microsegregation effects. Inclusion analysis, analysis of individual phases in multi-phase alloys, and investigations of denuded or enriched zones around grain boundaries and precipitates are all fields of enquiry which should benefit greatly from this new and non-destructive technique. In many such problems, however, it is the way in which a particular constituent or impurity is distributed throughout the microstructure that is of as much interest to the metallurgist as is the analysis at a few selected points. It is for this reason that the X-ray scanning microanalyser, developed originally at the Cavendish Laboratory, Cambridge,[3,4] and more recently at Tube Investments Research Laboratories,[5] possesses distinct advantages for many applications.

The instrument, when the scanning facility is not in use, functions as a static probe microanalyser similar to those developed by Castaing in France and Mulvey[6] in this country, though the latter instrument was later converted for scanning ; other static probe work is also reported from the U.S. and Russia. When the scan is switched on, however, deflection coils in the column scan the probe over a square raster on the specimen. The X-ray emission from a single element in the surface can be selected by means of a crystal spectrometer and is then detected with the aid of a proportional counter. The signal from this counter is used to modulate the brightness of a beam scanning a cathode ray tube in synchronism with the probe scanning the specimen. An image can thus be produced on the screen having point-for-point correspondence with the area scanned on the specimen and displaying semi-quantitatively the distribution of the chosen element over this field. The magnification can be controlled within the range 200–3,000 by varying the amplitude of scan on the specimen, while keeping that on the screen constant.

Some of the back-scattered electrons are also utilised in a similar manner, through the medium of a scintillation counter, to form an image of the surface. This image, which is displayed on a second cathode ray tube, is similar in contrast quality to an optical image photographed in oblique illumination and thus reveals topographical detail. Contrast is also produced by variations in atomic number in the surface, the more massive atoms having a greater tendency to back-scatter electrons. Compared with an optical image of equivalent magnification, this electron image has considerably greater depth of focus.

When scanning, the rate at which information about the specimen surface is collected is very high, although of course a sacrifice is made in the amount of quantitative information available about any single point.

Several different types of information are thus made available :—

(*a*) *From the Electron Image*—Topographical detail and information about atomic number variations in the surface.

(*b*) *From the X-ray Image*—The identity of elements present in the specimen surface and their distribution over the area scanned.

(*c*) *Linear Concentration Display*—This facility consists of a semi-quantitative display of the distribution of a selected element along a chosen line on the specimen and is made available by using one of the display tubes to monitor the X-ray emission as a Y-deflection while the probe is scanned slowly along the line.

(*d*) *Quantitative Analysis at a Point*—The probe can be positioned on the point to be analysed by reference to either the electron or X-ray images, which persist in the afterglow after the scan has been switched off. The precision with which this can be carried out is better than the diameter of the probe.

If the maximum amount of information is to be extracted from the specimen, the conditions under which the instrument is operated have to be optimised in favour of whichever type of information is required. Basically this involves adjusting the relationship between resolving power, emitted X-ray intensity, and recording

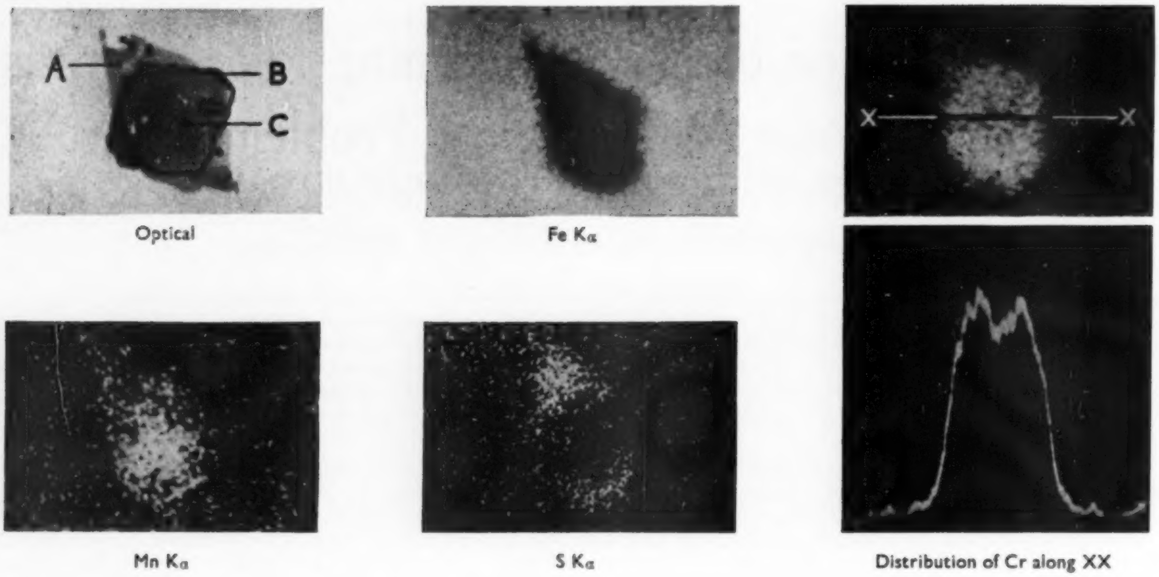


Fig. 1.—Duplex non-metallic inclusion in low alloy steel. ×**1,800**

time.[4] For example, achieving the highest resolution in the electron image ($<0 \cdot 5\mu$) requires a small probe diameter, resulting in a low probe current and hence in low X-ray intensity. On the other hand, since the probe diameter is not then the limiting factor so far as the resolution of the X-ray image is concerned, but rather the lateral diffusion of electrons within the specimen, it is better when forming the X-ray image to increase the probe diameter to about 1μ to match this spread. The resulting increase in X-ray intensity reduces the graininess or "noise," which tends to obscure contrast in the image due to the statistics of image formation with a finite number of X-ray quanta. In cases where the X-ray intensity is still too low to give a satisfactory image, the probe diameter can be further increased with some loss in resolution, or, alternatively, the field of scan narrowed to include only the feature of interest. The number of quanta recorded for each point scanned in this feature is thereby increased. The exposure time is normally set for convenience at 2 mins., but this can with advantage be increased several times to a point limited only by the stability of the apparatus. Similar considerations of resolution, intensity and recording time apply also to spectrum plotting from a selected point, where it may be necessary to measure accurately the height of a small characteristic peak against a background of continuous radiation.

The examples that follow illustrate the effect of adjusting these variables as far as possible to the optimum, but some noise remains and is visible in the X-ray images.

APPLICATIONS

Inclusion Analysis

Specimens of a steel containing 3·67% Cr were circulated to members' laboratories by the B.I.S.R.A. Committee on Gases and Non-Metallics as part of a programme of work designed to assess the performance of the many new physical techniques now available for inclusion analysis. The main interest in this case was in the fine dispersion of carbides ($<1\mu$ in diameter) produced by various heat treatments, but the material also contained slightly grosser inclusions of two types. One of these was a pale grey apparently plastic material, which occurred in the form of stringers greatly elongated in the rolling direction. The other was a darker grey, more rigid type of material which, if it deformed at all, appeared to fragment in a brittle manner, forming a shower of small pieces.

The particle selected for microanalysis had a characteristic diamond shape which made it easily recognisable, and examination under the optical microscope revealed that it was composed of both types of inclusion—a rigid particle roughly square in outline with a small amount of the pale grey material on either side. This had flowed in such a way as to form a diamond shape approximately 10μ in length with the major axis in the rolling direction. Duplex inclusions of this kind appear frequently in many types of steel.

X-ray images were obtained and photographed selecting Fe Kα, Cr Kα, Mn Kα and S Kα radiations (Fig. 1). On inspection and comparison of these photographs several facts are readily apparent.

(*a*) Iron is not a major constituent of either form of inclusion material, although the area of the rigid particle appears to contain more than the softer material.

(*b*) Chromium is a major constituent of both types of material and is distributed throughout the inclusion, with perhaps slightly less in the centre of the rigid particle than elsewhere.

(*c*) Manganese is present to a small extent thoughout, but in appreciable quantities in the darker, central region of the rigid inclusion.

(*d*) Sulphur is present only in the softer material at either end of the rigid particle.

The denudation in chromium in the centre of the rigid particle (in the area that appears to be richest in manganese) has been confirmed and demonstrated in a semi-quantitative manner by means of the slow scan facility. A photograph of this trace is reproduced showing the distribution of chromium along the line scanned and, in particular, the lower count rate obtained from the centre of the particle.

The information obtained from these X-ray images has been supported by the results of point analyses made in the positions *A*, *B*, and *C* indicated on the optical photograph.

Position *A* in the sulphur rich region analysed :—

Fe%	Mn%	Cr%	S%	Total %
8	2·5	43	44·5	98

The value obtained for the sulphur content was arrived at by comparison with a particle of MnS in a piece of mild steel, the assumption being made that this particle contained nothing else but Mn, Fe and S. This was probably quite justifiable but, owing to the poor transmission of S Kα radiation (λ=5·3Å) by the beryllium window of the proportional counter, the count rate was low and statistical errors were probably significant. The accuracy of these figures is probably not better than $\pm$ 2 or 3% but they do indicate the nature of the material, and the fact that no other major constituent is present.

Position *B* in the rim of the rigid inclusion analysed :—

Fe%	Mn%	Cr%	Total %
14·5	4	39	57·5

rather more iron and manganese than in point *A*.

Position *C* in the centre of the rigid inclusion contained

Fe%	Mn%	Cr%	Total %
13	10	36·5	59·5

a substantial increase in manganese compared to point *B* at the periphery and a decrease in chromium as indicated in the X-ray images.

In both these last two positions it is clear that a major constituent is missing and further searches revealed the fact that it was an element for which Z $\leqslant$ 14 ; the range of the spectrometer was subsequently improved to include Mg (Z=12) by substituting a flow counter with a thin Mylar window instead of the sealed-off counter used in this experiment. As silicon was not detected, the most likely possibility for the rigid particle is perhaps a mixed oxide of the chromite type, with manganese replacing some of the chromium towards the centre, over a region apparently corresponding quite closely to the darker zone in the optical image.

All the above quantitative results have been corrected for counting losses and also compensated for fluorescence and absorption effects. The absolute magnitudes of these corrections have usually been of the order of 2–3%. Microanalysis at a point in the matrix gave a value of 3·6% for the chromium content, the bulk chemical analysis value being 3·67%.

Surface Segregation in Mild Steel Leading to Hot Shortness

During the oxidation of mild steels containing "tramp" elements such as Ni, Cu, Sn, and As, selective removal of Fe from the surface by oxidation leads to the formation of a layer at the bottom of the scale enriched in these more noble elements. The performance of the Cosslett-Duncumb instrument at the Cavendish Laboratory in revealing and analysing these sub-scale enrichments[7, 8] was a major factor in stimulating the construction of the instrument at Hinxton Hall.

The segregation of Cu and Sn in this way is widely believed to have an adverse effect on the hot workability of the steel,[9] the effect being particularly marked when both elements are present together. This behaviour has been ascribed to the formation of zones at the surface of the metal so heavily enriched in these two elements that they are molten at the temperature of working. The influence of arsenic in this connection has not been so widely discussed, but since it is now possible to analyse these enriched zones with some degree of accuracy a better assessment of their probable melting point can be made.

The first four images in Fig. 2 illustrate the appearance of this sub-scale enrichment in Cu, Sn and As. They consist of an optical image and X-ray images taken with Cu Kα, Sn Lα and As Kα radiations selected. The specimen is a section through the surface of a heavily scaled billet of composition :—

C%	Si%	Mn%	S%	P%	Ni%	Cu%	Sn%	As%
0·1	0·07	0·51	0·049	0·018	0·14	0·20	0·059	0·037

Showing a markedly similar distribution, enrichment in all three elements can be seen in a line at the bottom of the scale and also within the scale itself. In particular, towards the outer surface of the scale there is a large, heavily enriched region. This would seem to indicate that as the scale becomes detached during the working process there is a possibility that it may carry away with it some of the enriched sub-scale material. The extent to which this occurs and the mechanism by which this material becomes included in the scale is not immediately clear and is a point worthy of further study. The concentrations that can occur in these enriched regions are illustrated by the 19% Ni, 12% Cu, 12% Sn, 2·8% As recorded from a point in the sub-scale in the field shown.

It is also possible to study this effect in plan by polishing the specimen parallel to the surface until a section is obtained from just below the level of the bottom of the scale. The last two images in Fig. 2, an electron and a nickel X-ray image, have been obtained from a specimen prepared in such a way. This section is from just below the level of the continuous sub-scale, and it can be seen clearly that Ni enrichment has occurred at the austenite grain boundaries, the specimen having been quenched after heat treatment. This sample was heated for 1 hr. at 1,000° C. in air to produce this effect, and is a piece of commercial mild steel of composition :—

C%	Si%	S%	P%	Mn%	Ni%	Cu%	Sn%
0·12	0·212	0·048	0·024	0·57	0·25	0·20	0·088

The problem of elucidating the mechanism by which these enrichments occur and their relative importance in leading to surface hot shortness is at present under investigation at Hinxton Hall. Preliminary indications are that the simple picture of the more noble elements being left behind while iron is preferentially removed by oxidation requires some modification.

The Segregation of Lead in a Free-Cutting Steel

This specimen was kindly supplied by Mr. F. B. Pickering of the United Steel Companies, the problem being to discover the location of lead rich regions in the microstructure. Microradiographic evidence had pre-

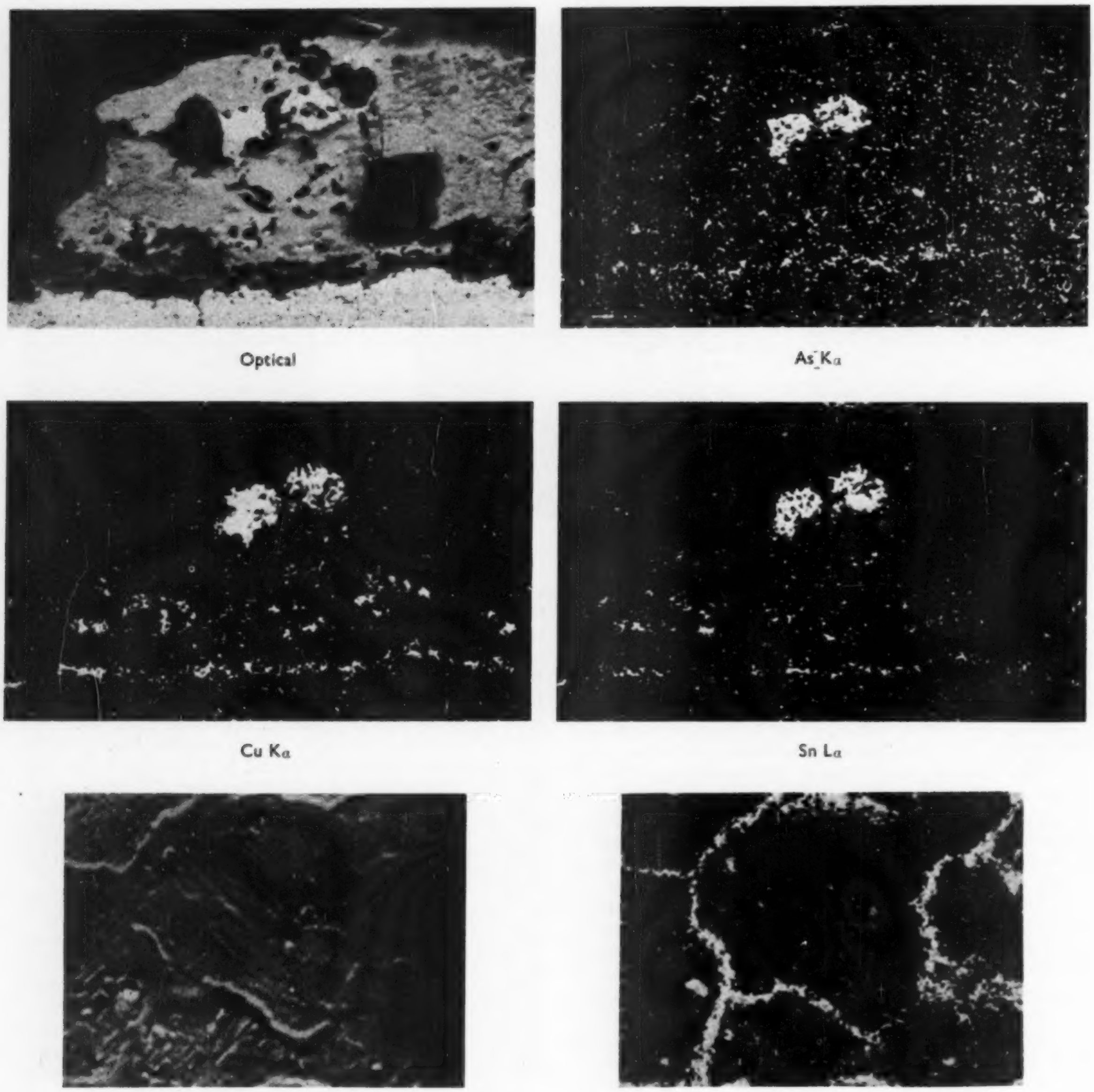


Fig. 2.—Segregation of " tramp " elements at the surface of mild steel. $\times$**210**

viously indicated that the lead tended to occur in association with manganese sulphide inclusions. Several of these inclusions can be seen in the optical image in Fig. 3 and also in dark contrast in the electron image. This contrast effect arises because the average atomic number of the inclusion material, manganese sulphide, is less than that of the surrounding iron matrix, and hence the electron back-scatter from these regions is lower. Associated with some of the inclusions in the electron image, very bright regions corresponding to intense back-scatter can be seen. These dark and bright contrast effects in the electron image, due to the local lowering or raising of the mean atomic number of the specimen by sulphur in the one case and lead in the other, should be compared with the X-ray images showing the distribution of these two elements. The electron image in this case is yielding information very similar to that obtainable from a surface microradiograph. In some instances this facility can be particularly useful, for example, in the study of inclusions and precipitates in beryllium. Most of the common impurities in beryllium are appreciably heavier than the metal itself, and thus stand out in very clear contrast in the electron image.

Analysis of Individual Phases in a Three-Phase Alloy

This specimen was kindly provided by Dr. D. R. F. West of Imperial College. It was prepared during the

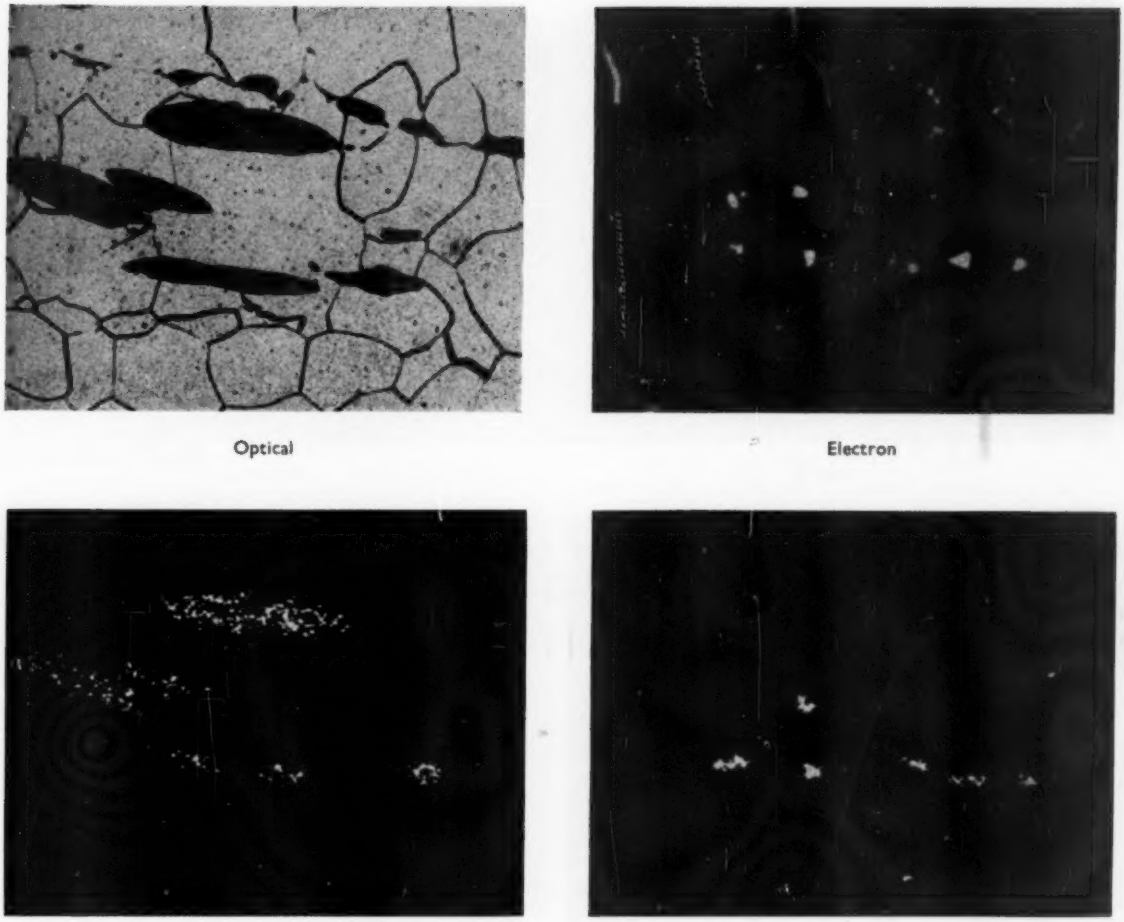


Fig. 3.—Lead and sulphur-rich inclusions in a free-cutting steel giving atomic numb er contrast in the electron image. ×450

course of an investigation into the mechanism of the pearlite-type eutectoid transformation in aluminium bronze. The composition of this material was

Cu%	Al%	Mn%
87·5	11·5	1

and it had been annealed at 800° C., isothermally transformed for one month at 551° C., and then quenched before examination. The transformation is nearly complete and the purpose of the examination was to determine the distribution of manganese between the α, β and γ phases at this stage.

The area chosen is a growth front of the well-developed pearlite structure, and a small island of residual β can be seen in the middle of the field (Fig. 4). Point analyses were carried out in the positions indicated by the black dots in the optical image. These are contamination marks that have been allowed to build up to illustrate the manner in which the probe can be positioned on each phase in turn. The diameter of these spots is, however, considerably greater than the probe diameter, as is evident from the resolution exhibited by the electron image. The greater topographical contrast as compared to the optical image is also worthy of note.

The values obtained for the manganese content of the three phases were :—

γ (lamellar material)	α (inter-lamellar material)	β
0·62%	1·16%	1·53%

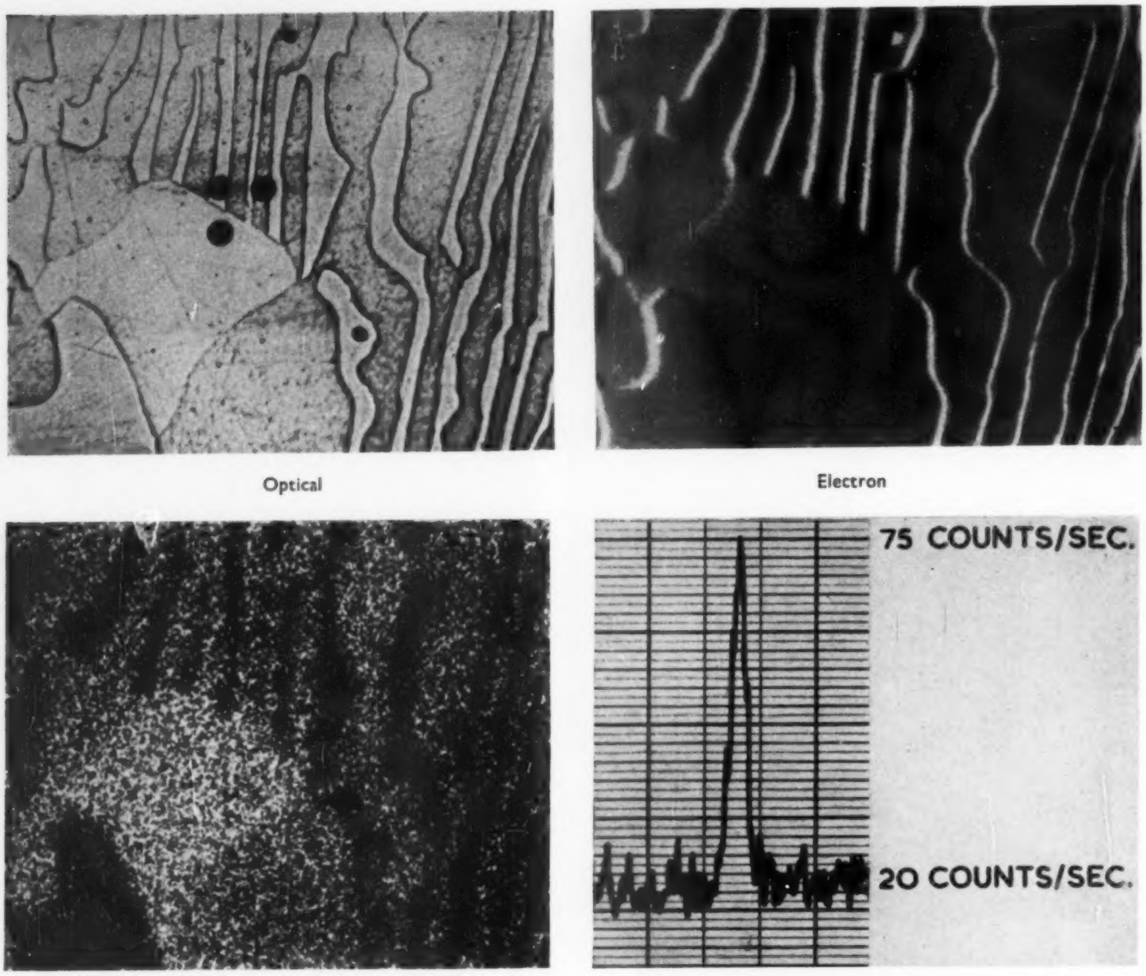
these are subject to a probable error of $\pm 0{\cdot}05\%$.

The pen recorder trace corresponding to the value of 0·62% Mn is also reproduced in Fig. 4, and shows a total of 75 counts above a background of 20, a peak-to-background ratio of nearly 4 : 1. It can be seen that under these conditions 0·1% of Mn would still be detectable, corresponding to a mass sensitivity of 10^{-14}g.

The X-ray image shown was obtained by using a probe current of 1μA. (approximately 10 times greater than the normal value) and a small sacrifice in resolution has probably been made. This does permit, however, the different Mn contents of the three phases to be distinguished. The exposure time was 4 mins.

Fracture Surfaces

The examination of fracture surfaces in the micro-



Optical | Electron

Mn Kα | Mn Kα from γ phase (0·62%)

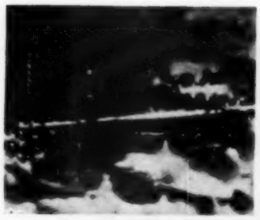
Fig. 4.—Distribution of 1% manganese in a three-phase aluminium bronze. $\times 600$

analyser is considerably facilitated by the comparatively large depth of focus of the electron image and the impression of surface geometry conveyed by the " oblique illumination " effect. Accurate quantitative analysis is difficult if not impossible under these conditions for several reasons. Firstly, the precipitates or other features of interest often stand slightly proud of the surface. Secondly, this surface is seldom normal to the incident electron beam and, even if it is, the X-ray emission may be partially absorbed by neighbouring features lying in the X-ray path to the spectrometer. Both these effects would lead to absorption and fluorescence conditions very different from those obtaining in the smooth surface of a pure standard, and thus invalidate the comparison of intensities required for a quantitative analysis. Thirdly, if the fracture is intergranular, the precipitates or denudation effects that are being studied may not extend far enough in depth for there to be any certainty that the whole of the probe is being absorbed within them. This again invalidates quantitative measurements involving comparison with bulk standards.

In spite of these limitations on quantitative analysis, much useful qualitative information can often be obtained. The example shown in Fig. 5 is a fracture surface of cast beryllium. This material has a comparatively large grain size and the cleavage faces are frequently undulating in nature. It was not possible to photograph this field in the optical microscope owing to the magnitude of these undulations. In the micro-analyser, however, the specimen has been rotated until as little as possible of the field is in shadow, and under these conditions a fairly large area can be examined in the electron image.

Several large precipitate particles can be seen on this surface and two of these have been examined at higher magnification. Both appear slightly dendritic in form but there is a marked difference in their general appearance. In one case (near the lower edge of the field) the side arms of the dendrite remain uniformly short along the length of the particle. Another particle lying closer to the centre has, in contrast, much longer side arms which diminish in length in a regular manner towards one end of the major axis.

From the X-ray images it is apparent that the first

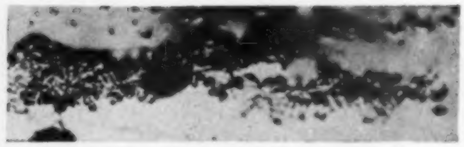


Optical

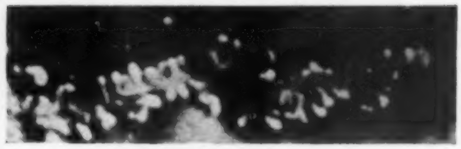
Electron

Ca Kα

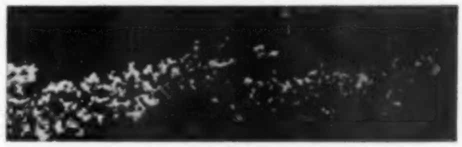
Electron



Optical



Electron



Ti Kα

Fig. 5.
Precipitates on a fracture surface of cast beryllium. Top centre photograph ×200; remainder ×400

type contains titanium and the second calcium. It was also established that the titanium particle contained no detectable calcium and vice versa. These images were obtained with an exposure of 6 minutes, the count rate from each particle being no more than 120 per sec. (compared with several thousand per second from the pure standards). This low value was in all probability due to the thinness of the particles and their comparative transparency to the electron beam.

This apparent correlation between the morphology and composition of co-existing precipitates is a type of information which can be very usefully fed-back into optical metallography. If the relationship is properly established these phases may then be recognised on sight without recourse to X-ray microanalysis, and in this way the technique can perhaps help to increase the scope and usefulness of the optical examination.

FUTURE DEVELOPMENTS

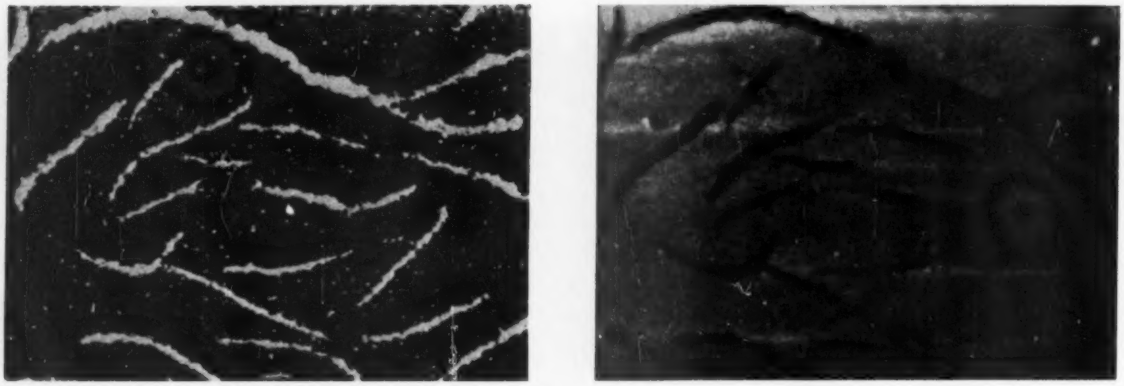
The Detection and Dispersion of Ultra-Soft X-Radiation

Apart from the limitation in resolution which is imposed by lateral diffusion of electrons in the specimen, the principal limitation of the method, as far as the metallurgist is concerned, lies in the difficulty of analysis for elements of atomic number less than 12. This problem is of course not exclusive to X-ray microanalysis, but applies to X-ray spectroscopy in general.

The lightest element which can be detected qualitatively and quantitatively by the present technique is magnesium ($Z=12$), or perhaps sodium ($Z=11$). This can be achieved using a mica or gypsum crystal to reflect the radiation into a proportional counter of the flow type, having a window of Mylar plastic film only 6 microns thick. Carbon, nitrogen and oxygen ($Z=6$, 7, and 8) appear to be a tantalisingly small distance below this limit. In this part of the periodic table, however, the wavelength of the characteristic X-ray emission increases rapidly. Although the K or L emissions of elements 12–92 all lie in the range 0·7–10·0Å, carbon K radiation has a wavelength of 44Å and thus its detection involves a fourfold increase in the wavelength range.

The difficulties lying in the way of such an extension, which exist both in the detection and dispersion of these very soft radiations, are not so insuperable as they once seemed. It has long been known that a diffraction grating spectrometer with photographic recording can be used in this wavelength range, but the low efficiency of the grating and the difficulty of making quantitative measurements render it inconvenient in this case. The problem hinges largely on improvements in technique and the availability of suitable window materials. The immediate cause of the practical limit at about $Z=12$ is that at longer wavelengths than this the absorption of a 6μ Mylar window (the thinnest readily available) becomes prohibitively great although, as Dolby[10] has shown, it is comparatively transparent again to carbon radiation (7% transmission at 44Å) which is on the other side of the carbon absorption edge. It thus acts to some extent as a filter discriminating against nitrogen and oxygen K radiations.

If a practicable and robust window could be prepared which transmitted all three radiations with adequate efficiency, the problem of discriminating between them would still remain, as the wavelength resolution of the proportional counter is not sufficient by itself to do this completely, and suitable crystals of a large enough



Carbon K — Electron

Fig. 6.—Graphite flakes in pig iron revealed by detection of carbon K radiation (44Å). ×200

spacing have yet to be found. It is here that the matrix method of pulse height analysis described by Dolby[11] would seem to offer considerable hope. This is essentially an electronic method of distinguishing the overlapping contributions to the pulse height spectrum of neighbouring elements, and has the advantage over other techniques that there is no loss of intensity before the radiation enters the counter due to the use of grating or crystal monochromator. The method has already been applied quantitatively in one instance[12] with success.

The practicability of detecting carbon K radiation and using it to form a scanning image is illustrated in Fig. 6. The specimen is a sample of pig iron containing a fine dispersion of graphite flakes. The X-ray image was obtained using a proportional counter of the needle type similar to that described by Duncumb.[13] The geometry of such an end-window counter enables it to be mounted close to the specimen and makes it possible to achieve a relatively high collection efficiency using a window of small diameter, which can thus be very thin. Under these conditions there is no shortage of counts and the accelerating voltage can with advantage be reduced to 8 kV., or less. At this voltage carbon K radiation is more than adequately excited while the background of continuous radiation is considerably reduced. With a suitable pulse analyser setting, a peak-to-background ratio of 6 or 7 : 1 can be achieved, so that one might expect to be able to detect 10–20% of carbon in this way. It should, however, be emphasised that much further work is needed before a reliable quantitative analysis for carbon is possible, either in the presence of oxygen and nitrogen or even by itself. Particular interest will of course attach to the technique when it can deal with carbon in amounts of less than 10 wt. %, since this is the range including most of the important metal carbides.

An important concomitant of any advance in this direction is the increase in resolution that is possible when working at very low accelerating voltages. This is due to the reduction in both the depth of penetration and the lateral diffusion of the electrons in the specimen. The improvement in resolution is accompanied by a decrease in the intensity of X-ray emission, which can be tolerated up to a point if improvements can be made in the efficiency of collection, and perhaps also in the efficiency of the electron gun. An analysis of the various limiting factors by Duncumb[13] suggests that a resolution of 1,000Å may be obtained in a scanning image, and that point analyses may be possible with even greater resolution if suitable counting techniques are employed.

As these advances occur the technique may be increasingly used in conjunction with electron as well as with light microscopy. In particular, the analysis of particles on extraction replicas should prove a most fruitful application. Even if absolute quantitative measurements are not possible under these circumstances, information about the ratio of constituents in mixed or non-stoichiometric precipitate particles would be very welcome.

ACKNOWLEDGMENTS

Thanks are due to Mr. F. B. Pickering, Dr. D. R. F. West, and the B.I.S.R.A. Committee on Intermetallic and Non-metallic Compounds for permission to include their specimens. The authors are also indebted to the Chairman of Tube Investments, Ltd., for permission to publish this paper.

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Changes

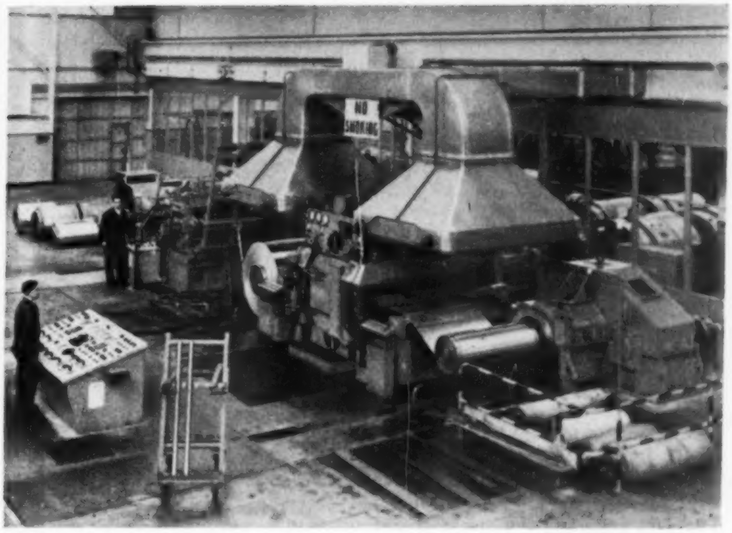
THE new London address of Brayshaw Furnaces, Ltd., is 232 Bishopsgate, London, E.C.2 (tel.: Bishopsgate 3575/6).

THE London office of Uddeholm, Ltd., is now at 124 Victoria Street, London, S.W.1. (telephones: VICtoria 6780 and 5711). The head office remains at Crown Works, Northwood Street Birmingham, 3 (CENtral 8971),

THE Incandescent Group announces that its London office telephone number has been changed from Sloane 7803 to Belgravia 7803-5.

Sendzimir Mill for Cold Rolling Aluminium Strip

Million Pound Installation at Birmetals' Woodgate Works



Aluminium alloy strip can be reduced from 0·1875 in. to 0·006 in. at a maximum speed of 1,600 ft. per minute.

WHEN, on Thursday, 7th April, 1960, the Marquess of Exeter, K.C.M.G., chairman of Birmid Industries, Ltd., formally set in motion at the Woodgate works of Birmetals, Ltd., the new Sendzimir mill for cold rolling light alloy strip it marked the virtual completion of a scheme decided upon in 1956, when a study of the trends in demand for aluminium alloy sheet revealed the need to embark on a programme of expansion. Some idea of the issues involved in making such a decision can be gained from the fact that the cost of the installation as a whole is almost exactly £1 million.

The mill itself—a 50 in. reversing unit—is believed to be the first of its kind in Europe to be used for rolling work-hardening high-strength aluminium alloys, and when in full production it will roll between 12,000 and 20,000 tons of aluminium sheet a year, depending on the final gauge. To obtain the best results from the mill, in terms of economic production, it is necessary to feed it with as long a hot-rolled coil as possible. The unit has therefore been designed to take a coil up to 10,000 lb. in weight, but the coil being rolled at the present time weighs just over 8,000 lb.

To instal equipment for casting and rolling a slab from which a single coil of this weight could be produced would have involved an additional capital outlay of some £2 million, and would have put out of work some of the existing plant. This was not considered an economical proposition, and ways were sought of overcoming the difficulty. One of those considered was the welding together of a number of smaller coils, and despite the many problems which had to be solved—not least the heavy short-period electrical demand—a scheme was formulated with the co-operation of a consultant and the welding machine manufacturer for the flash butt welding of strip up to 52 in. wide.

Many other items of equipment, all of which have had to have special attention to detail, have been incorporated in the whole scheme. These include a coil annealing furnace, a slitting line, a cut-to-length line, and a sheet annealing furnace.

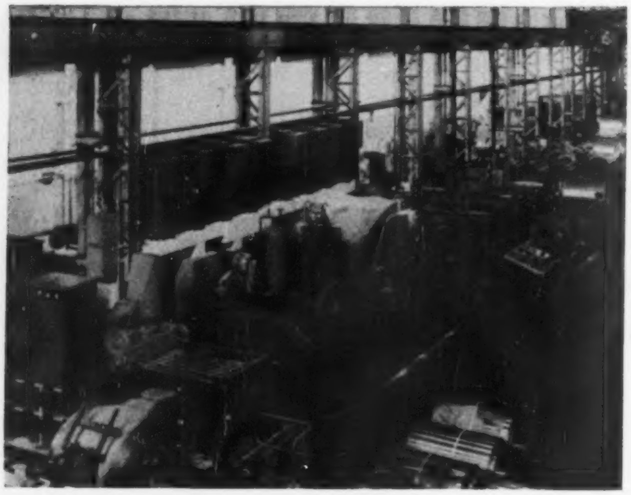
Slab Casting and Hot Rolling

The Woodgate works, which have been in operation since before the war, are devoted to the production in wrought form of a range of aluminium and magnesium alloys, and are equipped with melting furnaces, billet and slab casting equipment, and rolling mills and extrusion presses.

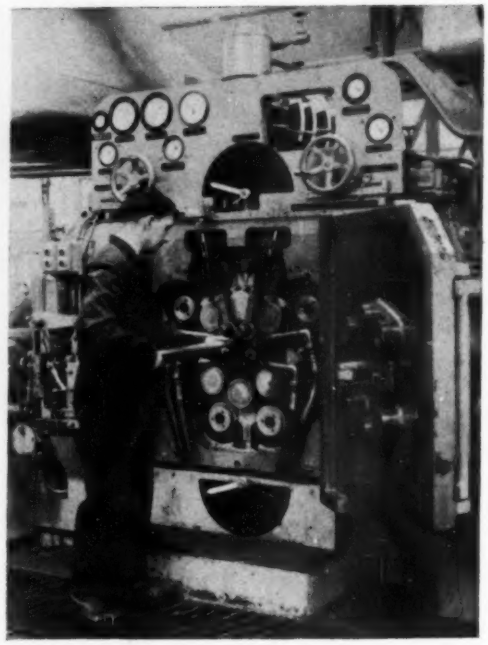
A line of 15-ton reverberatory furnaces serve as the main melting units for alloy preparation. They are fired by heavy fuel oil and have recuperators installed in the stack for preheating secondary air. Metal tapped from these melting furnaces flows by gravity into electrically heated (100 kW.) 8 ton capacity tilting holding furnaces, situated one adjacent to each melting furnace. In front of them are the machines for semi-continuous casting of billets up to 22 ft. 6 in. in length and 8 tons in weight. Multiple casting is used for smaller billets or slabs, and up to eighteen small billets are regularly cast simultaneously on one machine.

After casting, the billets or slabs are sawn to the lengths required by the rolling mills or extrusion presses. The products then go to the central metal stores, where they are checked and inspected for chemical analysis and surface quality before release for further processing. In the case of some of the larger billets and slabs, ultrasonic inspection is used as a means of detecting internal defects. Surface defects are removed by a scalping operation before the slabs are charged into one or other of the two 450 kW. electric furnaces, where they are heated to the temperature necessary for rolling in the breaking-down mill.

This mill has rolls 31 in. in diameter and 68 in. long, and provision is made on the run-out table at one side of the mill for the guillotining of thick plate. The other run-out table is equipped with a slitting machine, flying shears and an upcoiler, so that alloys can be cut into blanks for sheet rolling, or coiled in readiness for strip rolling according to requirements. Slabs up to 38 in. wide × 63 in. long × 9 in. thick are reduced in this mill to a thickness of $\frac{5}{16}$ in. before passing to subsequent mills.



The welding line comprises a pay-off stand, feeder leveller, shears, welding unit, weld dresser, edge trimmer, scrap chopper and coiling unit. The welding unit is situated on the left of the control panel.



Changing the work rolls on the Sendzimir mill. This operation can be completed in less than a minute.

Cold Rolling and Welding

Material intended for finishing in the Sendzimir mill is further reduced in thickness by cold rolling in an existing four-high strip mill which can produce finished strip down to a thickness of 0·022 in. in most alloys. This unit has 16 in. diameter × 69 in. long work rolls with 44 in. diameter back-up rolls. Coils from the breaking-down mill are annealed before entry into the four-high mill, where they are rolled to the required thickness with intermediate annealing where necessary.

The operations described so far are carried out on plant existing prior to the installation of the Sendzimir mill: from this stage onwards, the mill, furnaces, etc., form part of the " million pound plant," as it has come to be known at Woodgate works. All the new equipment—with one notable exception—is housed in a new building of the Portal frame type. The exception is the welding line, which is situated in the same building as the four-high cold mill. This is a convenient arrangement, as the purpose of the welding line is to join together four 2,000 lb. coils, from this mill so as to have one continuous 8,000 lb. coil for rolling in the Sendzimir mill.

The welding line comprises a Bronx pay-off stand; a feeder leveller; end shears; the A.I. butt welding unit; a Wadkin weld flash dresser; an edge trimmer and scrap chopping units by Auxiliary Rolling Machinery, Ltd.; and Wellman pinch rolls and tension reel. The first coil is passed through the line to the edge trimmer and has its rear end sheared about 3° off square. The second coil is then paid-off into the end shears and has its leading end sheared. The rear end of the first coil and the leading end of the second coil are gripped by the welding machine clamps at a distance apart fixed by a gauge. On operating the push-button control the ends of the coil are flash butt welded and forged, leaving an upset ridge or flash across the width of the strip. The flash is removed in the weld dresser before the strip is edge trimmed and the rear end of the second coil sheared ready for welding to the third. This process is repeated until the four coils have been joined together to produce a coil 50–53 in. in diameter and weighing up to 8,500 lb.

The key unit of the welding line is of course the flash butt welding machine designed and built by A.I. Electric Welding Machines, Ltd. Such an ambitious project as welding 52 in. wide aluminium alloy strip had not been attempted before, and it was fully understood by the company that the proposal was in many ways a gamble. However, as the company's technicians were confident that the fundamental idea was sound it was decided to go ahead. The results obtained—the outcome of co-operation between the company's technicians, the welding machine manufacturer and Mr. R. B. Giles, a consultant on the special problems of supplying electricity to the welder—have completely substantiated this confidence.

Birmetal's strip welder is reputed to be the largest of its type in the world, and for this reason alone the supply of electricity for this machine posed very unusual and difficult problems. Other inherent welder characteristics included an unbalanced load, extremely poor efficiency and an exceptionally short load period. The original design envisaged a current of 1,330 A. at 400 V. during the flashing period, and a current of 10,000 A. during the subsequent forging operation. Such a demand on the supply once in every five minutes was not acceptable to the electricity supply authority because of the interference to the supply to other users. Of three possible cures, one which had never previously been applied to flash butt welding machines—and in the opinion of many experts could not be so applied—was tried out on a small test plant at the manufacturer's works. On the strength of the success of these tests, the decision was taken to modify the welder, at a not inconsiderable cost, to enable this technique to be used. The results more than justify this step, since the loads have been reduced to approxi-

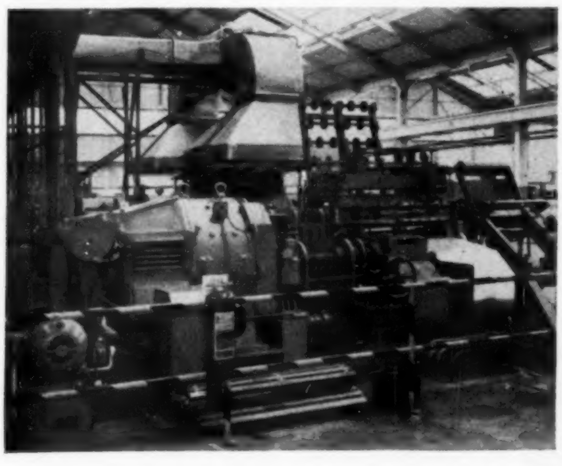
mately 725 A. (290 kVA.) during flashing and 5,800 A. (2,320 kVA.) during forging.

The Sendzimir Mill

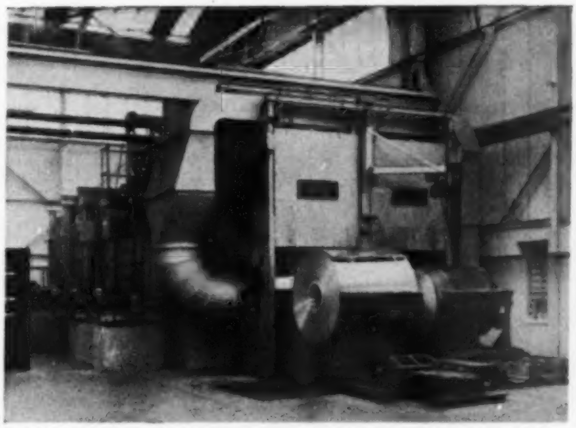
The Sendzimir mill is housed in a new Portal-frame building covering an area of 45,000 sq. ft. and conveniently situated to receive the coils from the welding line. The architect was Mr. F. J. Meeson and the civil engineering work was carried out by R. M. Douglas (Contractors), Ltd., and the steelwork by Braithwaite & Co., Structural, Ltd. The main bays are 200 ft. long by 78 ft. wide; one houses the mill and the other a Hallden-Robertson cut-to-length line and a G.W.B. flash annealing furnace. The despatch bay, which is 120 ft. long and 60 ft. wide, is connected to, and forms an extension of, the existing inspection and despatch department. Adjoining the mill is a 40 ft. wide bay containing a motor room, an oil filtration plant, a fitting shop, a sub-station, and a Stordy coil annealing furnace, which is charged from, and discharges into, the mill bay. The building is clad with Birmabright aluminium alloy sheet down to the 7 ft. 6 in. high perimeter wall. All roofing and side sheeting is lined with wall-board and insulated with 1 in. thick bonded Fibreglass: it is expected that the cost of the lining will be recovered in a little over three heating seasons.

Built by W. H. A. Robertson & Co., Ltd., the mill itself is a 50 in. reversing unit capable of rolling aluminium alloy in coil form at speeds up to 1,600 ft./min. The finished width of the coil obtained is 48 in. and the thicknesses which can be produced range from $\frac{3}{16}$ in. (0·1875 in.) down to as little as 0·006 in. The thickness on the outgoing side is indicated by a Baldwin nucleonic thickness gauge using Strontium 90 as a source. The work rolls are $2\frac{3}{8}$ in. diameter and each is driven by two back-up rolls, which in turn are backed by an arc of 3 rolls and a further arc of four rolls. There are thus twenty rolls in the mill, the arrangement being clearly shown in the illustration.

As in other Sendzimir mills, it is possible to change the actual work rolls in less than a minute, so that the last pass can be made with highly finished rolls which produce a mirror surface on the strip. Rolls are ground and polished on a Naxos-Union roll grinder supplied by Wickman, Ltd. A hydraulically operated form of crown adjustment is fitted which provides a range of camber control to the back-up assemblies and consequent control of strip shape. It is possible to adjust the camber to obtain absolutely uniform transverse gauge in the strip throughout the range of reductions and material tempers.



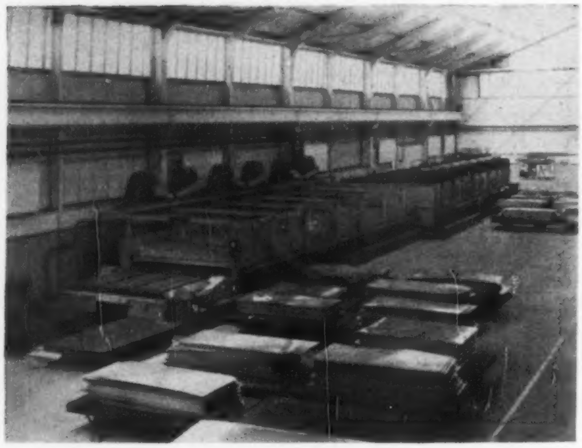
Entry section of the mill showing pay-off reel motor and gear box.



The coil annealing furnace will hold twelve coils of 8,000 lb. each when working in a continuous cycle.

The coils are fed through flattening rolls into the mill from a pay-off reel, and are then recoiled ready for passing through the mill in the opposite direction. As many as eight or nine passes may be necessary to reduce the thickness to the required value, with intermediate annealing where desirable. Hydraulic and lubrication supplies to the pay-off unit and feeder unit, and to the mill and coilers, are obtained from pump units and their associated tanks in an oil cellar which also contains the clean and dirty rolling-oil tanks, with a combined capacity of 16,000 gal. Rolling oil is supplied to the mill at 677 gal./min. at 150 lb./sq. in. pressure, and is thermostatically controlled to maintain its temperature. Cooling is effected by means of two Heenan and Froude water coolers in conjunction with two Serck heat exchangers. A by-pass filtration system developed by Birmetals maintains a clean supply of rolling oil which in this type of mill serves also as a lubricant to the mill back-up bearings. All motors in any way associated with the rolling oil are of flameproof construction and wired in Pyrotenax cable. The oil cellar is fitted throughout with automatically operated CO_2 fire extinguishing equipment.

Two coupled 900 h.p. 500/1,000 r.p.m. D.C. motors provide the main drive through David Brown gearing, but either motor can be used alone. Each coiler is driven by three 350 h.p. 500/1,250 r.p.m. D.C. motors, through gearing of the same make, to provide a maximum tension of 25,000 lb. By means of an electrically operated disengaging coupling the inboard motor may be used on its own to provide the lower tension values up to 8,000 lb. when lighter gauge material is being rolled. Since the strip tension is greater than in conventional mills, tension control must be accurate if uniformity of gauge is to be maintained throughout the length of the strip. Provision has been made for the automatic control of tension by the inclusion of load cells, one being fitted to each of the two deflector rolls, over which the tensioned strip passes. Flux-resetting magnetic amplifiers are used



Continuous nickel alloy tapes carry the sheets through the 40 ft. long flash annealing furnace and into the 42 ft. long cooling chamber.

for control of both coiler motor and generator, and in this way the need for rotating regulators is avoided. An 11 kV., 3,500 h.p. synchronous motor drives the D.C. generators, two of which are rated at 750 kW. and two at 870 kW., 600 V. The former supply the mill motors and the latter the reel motors. All the generators and motors were supplied by the Heavy Plant Division of Associated Electrical Industries, Ltd.

Cooling air is drawn through a Visco filter unit and discharged into a wind tunnel beneath the motor room by four 25 h.p. axial flow fans. This air is fed to all motors and generators, and the resultant air pressure built up in the motor room is controlled by electrically operated roof ventilators.

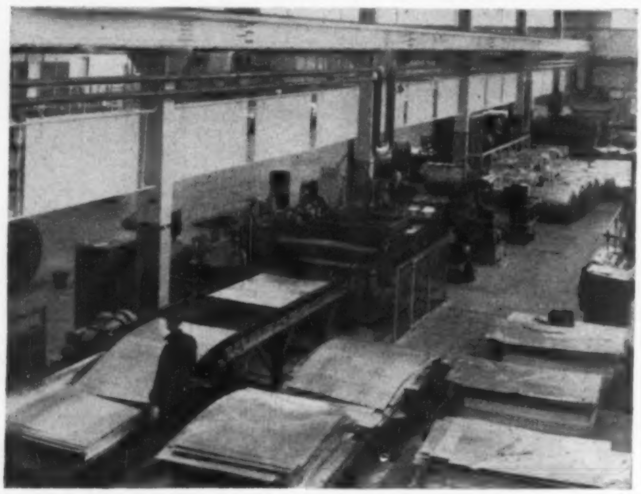
Annealing and Finishing

Intermediate annealing is carried out where necessary in the 550 kW. regenerative type coil annealing furnace designed to Birmetals' requirements and built by Stordy Engineering, Ltd. The furnace will hold twelve 8,000 lb. coils when working on a continuous cycle and has a heating capacity of 8,000 lb. an hour with an operating temperature of 450° C. It comprises a cooling chamber, three preheating chambers and the furnace chamber proper, each compartment being isolated from the adjoining ones by means of vertically rising insulated doors which have a motorised operation from an adjustable timer. Each coil is progressed on a stillage along an ingoing lane, through the cooling chamber and the three preheating chambers into the furnace proper. At the far end of this section it is traversed to the parallel outgoing lane and is then pushed in the reverse direction through the same chambers. In the cooling zone, fans blow cold air across the hot coils and towards the cold incoming coils. Similarly, in each preheating chamber, a specially designed air circulation system ensures efficient transfer of heat from the outgoing hot coil to the ingoing cooler one. In this way, the coils are preheated almost to annealing temperature before entering the furnace proper. Each preheating zone is provided with a 50 kW. boost heater designed to operate when the furnace is first being charged and when there are no hot coils available. The effect of these heat transfer arrangements is to reduce the electrical loading to a little over half that for orthodox batch heating.

From the Sendzimir mill, the finished coils pass to the cut-to-length line, which has a maximum strip speed of 350 ft./min. and a thickness range of from 0·080 in. to 0·010 in. Any length from 24 in. to 192 in. can be cut on this line, which is a Hallden unit made and supplied by W. H. A. Robertson & Co., Ltd., with motors and M. G. set supplied by The English Electric Co., Ltd. Provision has been made for a slitting line on which 52 in. diameter coils will be slit to any width down to 1 in. This line, by Auxiliary Rolling Machinery, Ltd., will have a maximum speed of 800 ft./min. and a thickness range of 0·080 in. to 0·005 in.

A flash annealing furnace, designed, manufactured and installed by G. W. B. Furnaces, Ltd., is located in the same bay as the cut-to-length line. It will take sheets up to 8 ft. wide, or two sheets of 4 ft. side by side, and has four heating zones with a total rating of 600 kW. Continuous nickel alloy tapes carry the sheets through the furnace, which is 40 ft. long, and its extension, in the form of a cooling chamber, which is 42 ft. long. Extensive tests were carried out during the commissioning stage to ensure the maximum uniformity of heating across the width of the material. The conveyor speed may be varied from zero up to a maximum of 100 ft./min.

Sheets leaving the flash annealing furnace are inspected on both sides before being passed to the despatch bay, and ultimately to the customer.



General view of the cut-to-length line which has a maximum strip speed of 350 ft. per minute.

Aluminium at Sub-Zero Temperature

Symposium Organised by Northern Aluminium

A SYMPOSIUM on " The Use of Aluminium in the Transport and Handling of Liquefied Gases at Sub-Zero Temperatures," organised by Northern Aluminium Co., Ltd., was held at Aluminium Laboratories, Ltd., Banbury on April 12th and 13th, and was attended by about a hundred representatives of the shipping, shipbuilding, chemical and petroleum industries, of fabricators and manufacturers, and of research organisations and government departments. The main purpose of the symposium was to present for discussion the results of research and investigation carried out in this field at Banbury and Geneva by Aluminium Laboratories, Ltd., the research organisation of the Alcan group of companies, during the last two years. This work was initiated at the request of Northern Aluminium Co., Ltd., in view of the increasing interest in low temperature engineering generally, associated particularly with proposals already implemented on a full scale trial basis by the North Thames Gas Board, for transporting liquefied natural gas from the oil fields for use as a domestic and industrial fuel.

Aspects Discussed

Three papers were presented at the symposium ; the first, " The Mechanical Properties at Room Temperature and at $-196°$ C. of some Aluminium Structural Alloys," by R. J. Durham, head of mechanical testing section (Banbury), discusses the mechanical properties of some aluminium alloys suitable for structural use at very low temperatures, with particular reference to NP 5/6 (Noral B54S) plate in thicknesses of up to 2 in. The resistance to crack propagation in both parent metal and welded joints has been measured by the U.S. Navy Tear Test at both room temperature and $-196°$ C. and has been found satisfactory. In presenting the paper Mr. Durham gave details of a specially devised " wide plate " test that had been carried out on a welded, notched test piece fabricated from 2in. plate, using the 4,000 ton plate stretcher at Northern Aluminium Rogerstone works, with the object of eliminating the storage of strain energy factor in the testing equipment that, under laboratory conditions, has obscured the results of some of the tests on the thicker gauges of material.

The second paper, " The Design of Aluminium Tanks for the Sea Transport of Liquefied Natural Gas " was read by W. Ferguson, structural engineer, of Geneva, and contains the results of investigations into the structural design of special inner tanks necessary in ships for the carriage of liquefied natural gas (methane). Such tanks must be designed to resist considerable static and dynamic loading, and must also satisfy certain exacting conditions as regards their arrangement and method of mounting within the hull. These problems are fully discussed in the paper and details of several alternative types of tank are presented. The dimensions of these have been selected with a ship of the 30,000-ton-tanker size in mind as offering the greatest design problem, but because the suggested approach to the design calculation is set out in detail, the method used can be applied to smaller tanks.

" Some Aspects of the Fabrication of Vessels for the Storage and Transportation of Liquefied Gases, with particular reference to the Welding of Thick Plate " was the title of the third paper, read by A. R. Woodward, head of joining division, (Banbury). After discussing briefly the relative merits of the tungsten-inert gas (T.I.G.) and metal-inert gas (M.I.G.) processes for aluminium welding, this paper deals with the use of conventional M.I.G. equipment for welding NP 5/6 plate in thicknesses of up to 2 in., and describes development work on the use of high currents for the welding of thick plate with a minimum number of passes. The economic importance of establishing realistic standards is emphasised and the author suggested that one useful purpose of the paper might be to stimulate discussion on this subject.

In addition to the papers read, appropriate demonstrations and exhibits were arranged as illustrations of the various subjects discussed. The plate used in the large scale tear test was also displayed. A full report of the proceedings and discussion during the symposium will be published by Northern Aluminium Company in due course.

Bulk Propane Supplies to Industry

DISTRIBUTION of bulk liquid propane, supported by free technical service and maintenance, is among the latest advanced facilities offered by British Oxygen Gases, Ltd., to large industrial customers throughout the United Kingdom. Bulk holding tanks, enabling liquid propane to be stored without loss, can now be installed on a free loan basis at customers' works. Already the new system is in use in steelworks, shipbuilding yards, scrap, demolition and shipbreaking yards, and engineering works. For more than sixteen years B.O.G., has built up a complete propane distribution service covering the whole of the United Kingdom. During that time the demand for propane has increased considerably, and its suitability as a fuel gas in steel cutting has become well established.

Industrial customers using large quantities of propane from cylinders are faced with problems of excessive cylinder handling and possible loss of factory floor and yard space. To overcome these difficulties, B.O.G., can offer either cylinder manifolds and pipelines with individual outlet points, or for the large consumers, bulk storage tanks and pipelines. Simplicity and convenience in handling and floor space, and economies in price, make the bulk propane system readily appreciated by industry.

Bulk propane holding tanks are offered to customers on a free loan basis. Large industrial users can change quickly from cylinder supply to the bulk system and enjoy more advantageous prices, with little or no capital outlay for the new installation. The new bulk propane installations are maintained free of charge by specialist personnel. Regular visits are made to customers in all districts. Speed and efficiency are safeguarded, and repairs where necessary are immediate and without charge.

United Steel Development Plane for Appleby-Frodingham and Samuel Fox

UNDER a new development scheme costing a total of £32½ million, Appleby-Frodingham Steel Co. plans to increase its productive capacity for iron and steel and to install a new rod/bar mill, and Samuel Fox and Co., Ltd., is to add 100,000 tons per annum to its steelmaking capacity. Both works are branches of The United Steel Cos., Ltd., and these major projects, which are scheduled for completion by the autumn of 1964, represent the largest development scheme ever to be undertaken by the company: Iron and Steel Board approval has been given. When completed, together with other schemes already in hand, the ingot capacity of United Steel will be raised from its present level of about three million tons to about four million tons per annum.

At present, Appleby-Frodingham has a planned annual productive capacity of slightly exceeding one million tons of plates and sections. By the end of 1964, however, with the addition of the output from the rod/bar mill, this figure will approach 1,400,000 tons of finished products per annum. The decision to install a rod/bar mill with an additional capacity of 300,000 tons per annum has been taken in order to diversify the company's output of finished products, and to extract the full potential from the existing primary iron and steelmaking plant at Appleby-Frodingham's Scunthorpe works.

This expansion will call for increases in output as far back in the production sequence as the initial raw materials. Thus, United Steel's Ore Mining Branch plans to increase the level of its ironstone extraction from 6,190,000 tons per annum to 7,700,000 tons per annum, and United Coke and Chemicals Co., Ltd., is to provide an additional 323,000 tons of metallurgical coke per annum by doubling the size of its Brookhouse coke oven plant and by adding 50% to the capacity of its Orgreave plant.

Appleby-Frodingham were pioneers in this country in the use of 100% sinter burdens for blast furnace operation, and to produce the necessary additional tonnages of iron, increased tonnages of sinter of improved quality are to be made available by extensions to the present ore preparation plant. Large scale trials, utilizing sinter of improved size and physical condition, have already yielded significant increases in iron production. The proposed extensions, together with the addition of certain blast furnace auxiliaries, including additional stove capacity, gas cleaning plant and boiler plant, will permit the pig iron output to be raised from its present level of 1,560,000 tons per annum to 1,950,000 tons per annum by the end of 1964.

Recently, Appleby-Frodingham announced the development of the Ajax oxygen process for the making of steel. This process was developed to utilize a conventional basic open hearth tilting furnace for oxygen steelmaking. The steelmaking capacity at present available in the company's melting shops consists of ten basic open hearth tilting furnaces and three mixers. In the main, the open hearth furnaces are each of 300 tons capacity Two of them have already been converted to the Ajax oxygen process and a third conversion is now taking place. Under the proposed development scheme, five more open hearth furnaces will be converted to the Ajax process while one open hearth furnace will be converted, by rebuilding, to a hot metal mixer.

The Ajax process is operated most efficiently on high percentages of molten iron rather than on molten iron together with internal mill scrap. In consequence of these conversions, therefore, large quantities of virgin mill scrap, arising from the company's mills, will be released for use in the new electric arc furnaces which are to be installed as the principal feature of a £10 million scheme recently announced by Steel, Peech and Tozer at Rotherham.

So that the development programme can be implemented without reducing ingot production, it will be spread over a period of four years. Thus, ingot capacity will be raised progressively from its present level of 1,500,000 tons per annum to 1,900,000 tons per annum. When completed, these plans for additional ore mining, coke production, ironmaking and steelmaking, will yield an extra 400,000 tons of ingots per annum, of which 350,000 tons will be required for the operation of the new rod/bar mill. The remaining 50,000 tons are expected to be absorbed by the expanding production of existing rolling mills.

Surplus capacity for the rolling of blooms already exists in Appleby-Frodingham's cogging mill, which serves the section mills, and it is proposed to make this surplus capacity up to the equivalent of 350,000 ingot tons per annum by the installation of a four-strand continuous casting plant for the production of the remainder of the necessary blooms. The continuous casting plant is to be designed and manufactured by Distington Engineering Co., Ltd., a subsidiary of United Steel, which recently established a continuous casting division.

The blooms—the primary raw material for the rod/bar mill—will be rolled into billets in a new 32 in. reversing mill housed alongside the rod/bar mill. These billets will be the secondary raw material for the rod/bar mill. It is expected that the bulk of the mill's production will be in coils of up to 1,000 lb. in weight in a size range from 5 gauge to 1¼ in. in diameter. It is intended, however, to sell a quantity in bar form, and length straightening machines are to be installed for this purpose. The layout of the new mill will be designed to allow for the later addition of a bar cooling bed and its associated equipment.

It is estimated that several hundred additional jobs will be created at Appleby-Frodingham as a result of this development scheme.

At Samuel Fox, redevelopment of existing steel melting and rolling capacity will permit output to be increased from its present level of 400,000 tons to 500,000 tons per annum. This will enable the company to meet the increasing demand for its alloy and stainless steels.

The level of capital expenditure by United Steel will increase during the period of these schemes, although additional outgoings in the current year will not be very substantial and should be well within the scope of the company's existing resources. While retained profits are expected to make a considerable contribution to the cash requirements of the later years, additional finance will have to be raised in due course.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NEWS AND ANNOUNCEMENTS

Corrosion in Gas Plant Symposium

A SYMPOSIUM, jointly sponsored by the Institution of Gas Engineers, the Society of Chemical Industry Corrosion Group and the College of Advanced Technology at Birmingham, is being held at the College of Advanced Technology, Gosta Green, Birmingham, 4, on 22nd and 23rd, September, 1960.

It is anticipated that the meeting will be of value not only to those directly engaged in the gas industry but also to those working in allied industries. The topics to be discussed include fundamentals of corrosion and protection, organic protective coatings, the protection of structural steel against corrosion, the corrosion of cast iron in the gas industry, principles and applications of cathodic protection, protection of gas service pipes and fittings, corrosion and the gas consumer, and sprayed metal coatings in the gas industry.

Attendance is open to all. Those taking part will receive preprints of all papers which, at the meeting, will be taken as read. The registration fee is two guineas for members of the Institution of Gas Engineers, the Society of Chemical Industry, and *bona fide* students of universities and technical colleges. For others the fee is three guineas. Further particulars can be obtained from Mr. A. S. Blower, Department of Chemistry, College of Advanced Technology, Suffolk Street, Birmingham, 1.

A.D.A. Meeting

AT the annual general meeting of the Aluminium Development Association on Friday, April 29th, 1960, at 33 Grosvenor Street, London, W.1, the retiring president, Mr. Rudolf Hahn, presented the annual report. He noted that the continuing progress of the Association had been very satisfactory, with much work completed on the new three-year programme initiated during the year under review. Despite difficulties, such as the printing dispute, the general level of activity showed a further increase over previous years, and the high standing of the Association was acknowledged both at home and abroad.

Mr. W. Brining was elected president of the Association for the period 1960-61. Mr. Brining represents the Almin group of companies on the A.D.A. Council. He joined the Almin group as secretary on its formation in 1945, having previously been the secretary of International Alloys, Ltd., and became a director of Almin, Ltd., in 1952. Mr. Brining is also a director of six of the subsidiary companies of Almin, Ltd. Mr. R. Hahn, who represents the Association of Light Alloy Refiners and Smelters, Ltd., on the A.D.A., was elected vice-president for the ensuing year.

Welding Technology Courses

COURSES organised by the School of Welding Technology for the early part of next winter's session include: welded pressure vessels; residual stresses and stress relief; practical control of distortion; and metal spraying. The first of these will be held at the end of September and the remainder in October. Provisional arrangements are being made for further courses to be held in the period November 1960 to May 1961. These include welding of atomic energy plant; advanced course for welding engineers; practical ultrasonic inspection; design for welding in thermoplastics; inspection and testing; welded design and construction in general and mechanical engineering; brazing technology and design; welding for junior management; welded structures; and ultrasonic inspection. Further particulars may be obtained from the School of Welding Technology, 54 Princes Gate, Exhibition Road, London, S.W.7.

I.C.I./Alcoa Acquire Almin

IMPERIAL CHEMICAL INDUSTRIES, LTD., for themselves and on behalf of the Aluminum Company of America (Alcoa), have made an offer for the issued share capital of Almin, Ltd., the parent company of the Associated Light Metal Industries Group, which has been accepted by over 90% of the shareholders and is now unconditional. The Almin group comprises International Alloys, Ltd., Southern Forge, Ltd., Warwick Production Co., Ltd., Pressoturn, Ltd., and Aero Controls, Ltd. It includes the Fulmer Research Institute, which is an independent institution for carrying out sponsored research.

The group is an integrated and balanced organisation embracing a wide range of light metals technology, more especially aluminium, including secondary metal and finished products. The total assets—about £5 million—will make a notable addition to the I.C.I./Alcoa collaboration in the aluminium industry in Britain. This was initiated last September by the formation of the jointly owned company Imperial Aluminium Co., Ltd., and was further expanded recently by the acquisition of Invicta Foil Co., Ltd. and the rigid aluminium foil division of the Prestige Group, Ltd.

Paint Protection Course

THE Seventh Summer School on Corrosion organised by the Battersea College of Technology will be held at the College from 26th to 28th September, 1960. This course of nine lectures will deal with protection of metals by paints, with particular emphasis on recent developments in this field. The lectures, which will be given by authorities in their particular field, will deal with pretreatment; phosphating; mechanism of protection; paint formulation; paints for industrial, atmospheric and marine application; coatings for protection of metals underground; lacquers. Ample opportunity will be given for questions and discussion. The fee for the course is £10 10s. (inclusive of luncheon and morning and afternoon refreshment). Early application is advised: enrolment forms should be obtained from The Secretary (Summer School), Battersea College of Technology, London, S.W.11.

Ore Investment Corporation

A NEW company, British Ore Investment Corporation, Ltd. (B.O.I.C.), with a share capital of £5 million, has been formed by nine of the principal British steel-making companies using imported ore, and by British Steel Corporation, Ltd., an investment-holding company associated with the British Iron and Steel Federation. The main object of B.O.I.C. is to hold investments in overseas ore-mining companies—a function formerly

undertaken for the British steel industry by British Steel Corporation alone. The principal shareholders in B.O.I.C. are: British Steel Corporation Ltd.; The Steel Company of Wales, Ltd.; Colvilles, Ltd.; South Durham Steel & Iron Co.; Dorman Long (Steel), Ltd.; John Summers & Sons, Ltd.; Richard Thomas & Baldwins, Ltd.; Consett Iron Co., Ltd.; Guest Keen Iron & Steel Co., Ltd.; and The United Steel Companies, Ltd.

Amongst other investments, B.O.I.C. has taken over British Steel Corporation's 20% interest in Société des Mines de Fer de Mauritanie (M.I.F.E.R.M.A.), a £60 million project in French West Africa whose capital, equivalent to about £19 million, is being subscribed together with French, Italian and German interests. The ultimate intention of M.I.F.E.R.M.A. is to produce 6 million tons of iron ore a year. The ore will be transported on a railway, to be specially constructed, over a distance of about 400 miles to a port to be built near Port Etienne, in Mauritania. B.O.I.C. has also taken over the 30½% British participation in Compagnie Minière de Conakry, a company mining ore in the Republic of Guinea.

Metal Physics Lecture

Professor Linus Pauling, of the California Institute of Technology, has accepted an invitation from the Metal Physics Committee of the Institute of Metals to give a lecture during his forthcoming visit to Britain. His subject will be "The Structure of Metals and Intermetallic Compounds," and the lecture will be delivered at the Royal Institution, Albemarle Street, London, W.1, at 6.30 p.m. on Monday, 25th July, 1960. Visitors will be welcome; tickets are not required.

Laboratory Equipment Exhibition

The first Laboratory Apparatus and Materials Exhibition will be held in the Royal Horticultural Society's New Hall, Westminster, from 20th to 23rd June, 1960. Some sixty exhibitors will be showing laboratory furnishings, scientific apparatus, chemicals and glassware. Associated with the exhibition will be a series of lectures on various laboratory techniques, further details of which may be obtained from the organisers, U.T.P. Exhibitions, Ltd., 9 Gough Square, London, E.C.4. As seating accommodation is limited, early application for tickets is advisable.

Furnace Company Merger

The headquarters of The Furnace Construction Co., Ltd., have been moved to Normanhurst Chambers, 21 St. James Road, Dudley, Worcs. The move coincides with an amalgamation with Fuel and Metallurgical Processes, Ltd., suppliers of recuperators and hot blast equipment to the furnace and foundry trades. Mr. A. H. Preedy remains managing director of The Furnace Construction Co., Ltd., and Messrs. C. A. Bone, D. H. Evans, J. F. Sadler and Dr. G. J. Shaw have joined the board.

Castings on Show

At the Golden Jubilee Industrial Exhibition—organised by the North Staffordshire Chamber of Commerce at Hanley Park, Stoke-on-Trent, from 18th May to 4th June—samples of precision machined parts and non-ferrous and high duty iron castings are displayed by T. M. Birkett, Billington & Newton, Ltd., one of the largest non-ferrous founders in the U.K. Castings ranging from a few ounces to 10 tons are produced in a variety of non-ferrous alloys, including phosphor bronze, gunmetal, aluminium bronze, manganese bronze and light alloys. Specialities of the company include high tensile aluminium bronze castings; centrifugally cast wheel blanks; shell moulded castings; chill cast rods and tubes in 12–24 in. lengths; continuously cast rods and tubes up to 12 ft. lengths; and high duty iron castings up to 25 tons in weight.

New Agreement

Foundry & Metallurgical Equipment Co., Ltd., have made arrangements with Benno Schilde, A.G., of Bad Hersfeld, Germany, to manufacture and to market in Gt. Britain and Ireland, drying equipment operating on the well-known Benno Schilde System. The equipment includes uniflow type gas and oil fired sand driers of 1 to 10 tons per hour capacity; gas and oil fired portable mould driers; and batch and continuous core and mould drying stoves, including vertical tower and horizontal tunnel type driers. The equipment operating on the Benno Schilde System is specially designed to meet present day requirements in the foundry. Powerful fans and blowers are used in conjunction with special burners with full automatic temperature regulation to ensure thorough and uniform drying. The Benno Schilde portable mould drier is offered as an alternative to the Efco-Brown Boveri mould drier when electricity is not available.

Ether Langham Thompson (Italiana), Ltd.

As the first step in a policy of expansion in the export field, Ether Langham Thompson, Ltd., announce the formation of a new subsidiary company, Ether Langham Thompson (Italiana), Ltd., with offices and factory located at Via Bisleri 19, Milano, Italy. This new company will market and manufacture products of the subsidiary companies of Ether Langham Thompson, Ltd., namely: Ether, Ltd., J. Langham Thompson, Ltd., Electro Methods, Ltd., Datum Metal Products, Ltd., and Automation Systems & Controls, Ltd. In addition, agency agreements have been completed between the Italian company and a number of other well-known British and international companies engaged in the field of instrumentation.

Furnace Order

The Incandescent Heat Co., Ltd., Smethwick, has been awarded a major contract for the new Spencer steelworks of Richard Thomas & Baldwins, Ltd., at Llanwern, near Newport, Mon. The contract is for coil annealing plant with an output of 16,500 tons of 60-in. wide strip a week (nearly 100 tons an hour). In all, thirty furnaces will be supplied, of single and four-stack designs, together with furnace bases, forced cooling hoods and other ancillary equipment. The furnaces are the well-known Incandescent radiant-bowl fired type, such as have been supplied to leading steelworks in Britain and abroad. The complete contract, which is worth more than £1¼ millions, is believed to be the largest single order for steel sheet annealing plant ever placed in Great Britain.

Dielectric Heating - 2

The ability of dielectric heating to generate heat through the mass of a suitable material provides the following considerable advantages over other heating methods.

1 A body of uniform section and composition is raised in temperature uniformly throughout. Hence there is no waiting for heat to be transferred from an external heat source to the surface of the body and thence to its interior, and this is of particular advantage when the body is thick, and, as is often the case with dielectric materials, has poor heat-conducting properties.

2 The rate of heating of such bodies is, therefore, much faster than by external heating methods.

3 Since there is no external heat source, overheating or burning of the surface of a heat-sensitive material is avoided.

4 High thermal efficiency is achieved.

5 The amount of heat generated in the work is usually predictable and power input and heating time can be under positive control.

6 Production can start immediately after switching on and no current is used, nor heat lost, during periods of shut-down.

7 Vastly increased productivity is obtainable with less labour (usually unskilled), and fewer machines and less floor space are required.

8 Flexibility of layout makes it possible to plan the factory to best advantage, as dielectric heating equipment can usually be inserted directly into the production line.

Dielectric heating: typical application data

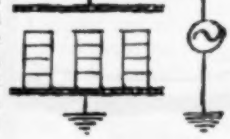
Typical application	Frequency	Radio frequency power
Thermoplastic welding.	20-100 Mc.p.s.	Up to 1 kW
Plastic pre-heating, wood glueing.	10-40 Mc.p.s.	2-30 kW
Plywood manufacture.	2-10 Mc.p.s.	Above 30 kW

Note: 1 Mc.p.s. = 1,000,000 cycles per second.

A few of the industrial applications of dielectric heating are briefly described below.

Preheating Thermosetting Plastics

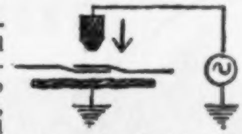
Dielectric heating is the ideal way of preheating moulding powder pellets used in the production of thermosetting plastic mouldings, since these materials are generally poor heat conductors. Properly applied, dielectric preheating promotes faster curing and hence a shorter moulding time, often increasing production ten to fifteen times. There is a marked reduction in tool wear, and thicker sections can be moulded, as the material is plastic when placed in the mould. There is less damage to, and movement of, metal inserts.

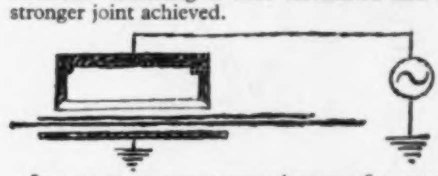


Welding of Thermoplastic Materials

An important and extensive application of dielectric heating in the plastic industry is the welding of thermoplastic sheets in the fabrication of such commonly used articles as raincoats, hoods, handbags, pouches and packaging materials. Dielectric heating is the only method which can usefully be employed since the heating electrodes, and hence the outside sheet surfaces remain cool while the inside surfaces forming the joint are fused, and a perfect weld results.

Two or more thermoplastic sheets are welded under pressure from electrodes suitably shaped to the area of weld required, the current being switched on at the same time as pressure is applied, and off as soon as the weld is completed and the pressure released. Stitching is thus eliminated and a far stronger joint achieved.





In most cases, component pieces are first cut from patterns and preliminary welding carried out to attach any fastening tabs and the like. The pieces are then brought together in a suitable loading frame and the main welding carried out to produce the complete article. In some cases, a suitably profiled electrode can be fitted with a knife edge to cut the sheets immediately outside the weld line, welding and pattern cutting being thereby carried out in the one operation. Dielectric welding can be applied also to very large thermoplastic products such as linings for swimming pools, and cinema screens.

Drying

Drying of materials by dielectric heating has the great advantage that the material tends to dry out from the centre, the reverse of what happens when external heating methods are employed, and the risk of overdrying and overheating of the surface is eliminated. In general ½ to 1 unit of electricity is required to drive off 1 lb. of moisture, depending upon the thermal properties of the material being dried. While the removal of large amounts of water from inexpensive commodities may sometimes be uneconomical, dielectric heating in the production line often leads to a higher overall production efficiency. It is valuable for removing final moisture traces and becomes increasingly economical as the value or heat-sensitivity of the commodity increases.

Rubber

External heating tends to dry and cure the surfaces of a thick latex mass before its centre, but dielectric heating properly applied promotes uniform conditions throughout.

Loaded rubber may not heat uniformly in a dielectric field due to uneven dispersion of its load, but nevertheless rubber preforms loaded up to about 15% are preheated dielectrically to reduce moulding times appreciably, the temperature evening out in the mould to give uniform curing.

Further examples are given in Data Sheet No. 12.

CUT HERE

RECENT DEVELOPMENTS

MATERIALS : PROCESSE : EQUIPMENT

Self-Balancing Electronic Temperature Indicator

THE latest Bikini instrument to be announced by Fielden Electronics, Ltd., is a precision self-balancing electronic temperature indicator designed to operate with resistance bulbs, but the use of transistors and modern production methods enable the instrument to be offered at a price claimed to be comparable with filled systems or galvanometer equipment. The indicator is housed in a sealed case similar in size and appearance to a circular scale electrical meter.

Resistance bulbs are used as the temperature sensitive elements and, with the inexpensive cable specified, these can be any distance up to 300 ft. away from the instrument without affecting the calibration or accuracy. This has been made possible by a newly developed measuring circuit. No trouble is experienced due to mains frequency pick-up on the leads to the resistance bulb since the circuit is operated at approximately 2,000 c./s. No moving coil, or other fragile assembly is incorporated, and the indicating pointer is precisely positioned by a simple servo-mechanism. Motive power is provided by a D.C. electric motor which has sufficient torque to ensure that the smallest temperature changes are faithfully followed.

A number of standard ranges, both Centigrade and Fahrenheit, are offered, within the normal limits of resistance bulbs (−200° C. to +500° C.) but non-standard ranges with supressed zeros and covering temperature spans down to 50° C. can also be supplied.

The instrument has a calibration accuracy of 0·5% of range, and reproducibility is better than 0·25%. It is, therefore, particularly suitable for those industries where high accuracy, long term stability and trouble free service are prime requirements.

Fielden Electronics, Ltd., Wythenshawe, Manchester.

Air Blast Tunnel Burners

" B-W " tunnel burners have been designed to meet the demand for faster and more efficient process heating to keep pace with increased production, and improvement in heating rates can be quite spectacular due to the high velocity and high temperature of the discharge gases. They are ideal for many direct local convection heating applications such as in forge work, billet heating and glass polishing, apart from installation in boilers, furnaces, ovens and similar industrial plants employing the principle of jet recirculation. The burners are of compact design incorporating their own combustion chambers and using air blast injection from a simple centrifugal blower to entrain gas at atmospheric pressure.

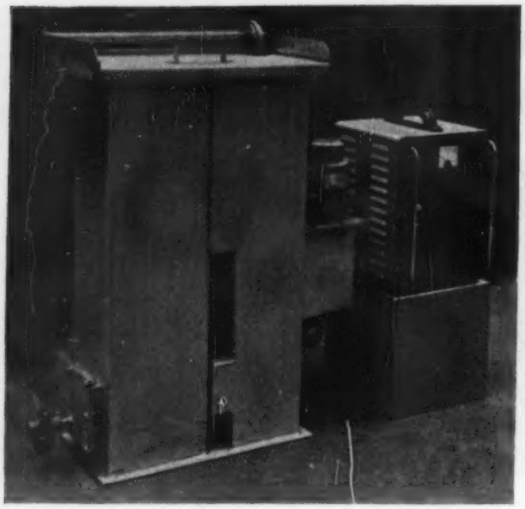
Initially, two standard types have been developed utilising research data made available by the Gas Council's Midland Research Station. Series " B " circular discharge models have metal-cased combustion tunnels for low and medium temperature applications (e.g. to 500° C.), and Series " C " slot discharge models for high temperature applications (e.g. 1,500° C.) have

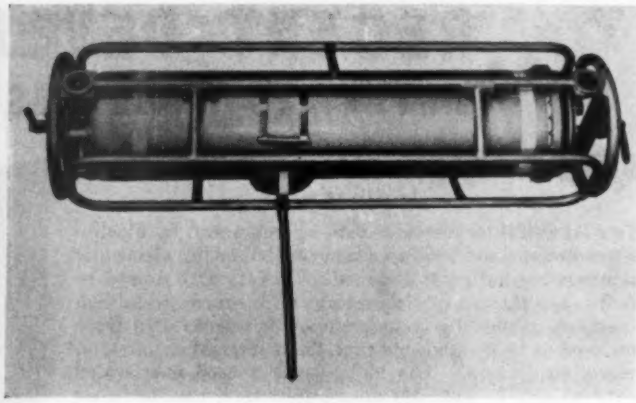
refractory cased combustion tunnels suitable for setting in brickwork. Capacities range from 0·5 to 10·0 therms per hour on town's gas (475 B.T.U. calorific value) when operating on 15 in. w.g. air pressure. Larger and special models and units for use with butane and other gases can also be supplied.

Barlow-Whitney, Ltd., 2, Dorset Square, London N.W.1.

Ultrasonic Degreasing Plants

I.C.I. is now marketing a series of ultrasonic cleaning plants designed for use with trichloroethylene. The smallest and simplest of these is the ULVI (compartment size 9 × 7 × 7 in. deep). Ultrasonic generating equipment, made by Dawe Instruments, Ltd., 99 Uxbridge Road, London, W.5, is connected to a sealed stainless steel transducer unit containing barium titanate elements submerged in cool trichloroethylene in one compartment of the plant. Here, electrical impulses at a frequency of 40 kc/s are changed to mechanical vibrations which produce " cavitation " in the solvent. Cavitation means the alternate formation and violent collapse, at a very high rate, of minute cavities in the liquid, giving an intense scouring action at the surfaces of the work. The high solvent power of trichloroethylene on oil and grease contamination is assisted by the mechanical effect of the scouring, so that insoluble solid particles are effectively detached, even from cracks and pits on metal surfaces. The solvent in the ultrasonic cleaning compartment is circulated by a pump through a filter, where solids are collected, and back to the same compartment : the filter is easily accessible for renewal. The other compartment in the plant holds electrically heated boiling trichloroethylene below a vapour layer. Vapour rises to the condensing coils, falls as liquid into the ultrasonic compartment and returns to the boiling liquor compartment by over-flowing the weir plate. A water coil prevents the temperature in the ultrasonic compartment from rising above 60° C. and damaging the transducers. The route of articles through the plant varies according to their condition. Most of the work for which the ULVI is appropriate is not heavily greased or oiled and can go straight into the ultrasonic cleaning compartment, and then into the vapour above the boiling liquor. Grossly





contaminated work should be given a preliminary dip in boiling liquor ; in the larger plants three compartments are provided so that a steady flow of work can be maintained. The final immersion in vapour is desirable for safety and economy, as it ensures that work is dry when it leaves the plant. Safety features include rim ventilation equipment and a thermostat which cuts off the heat if the water fails or if the solvent level falls dangerously low.

Imperial Chemical Industries, Ltd., Imperial Chemical House, Millbank, London, S.W.1.

Portable X-Ray Unit

A NEW lightweight, portable industrial X-ray unit of 300 kV. capacity, known as the Baltospot G300D, has been introduced by Pantak, Ltd., for examination of mild steel of up to 3 in. thickness. Both insulation and cooling of the X-ray tube in this unit is effected by a chemically and physiologically inert and non-inflammable gas. The use of this medium results in a lightweight tank head construction and achieves a high power/weight ratio, the weight being 142 lb. and the capacity being 300kV. The unit can be supplied with either a conventional 40° solid angle beam insert tube with a fine focus, size 3·0 mm. or a 360° circumferential radiation insert tube. Cooling of the anode is achieved in either case by forced circulation of the insulating gas by means of an internal blower.

The tank head, which is cylindrical in shape, is pressurised and is fitted with a pressure gauge so that the insulating gas may be checked at any time. A cradle is supplied in which are incorporated anti-shock mountings. The tube is protected against excessive temperature by a thermo-switch mounted in the tank head. A further device in the control unit simulates the thermal capacity of the anode and gives the operator a visual indication of overheating. A particular advantage is that a series of exposures can be planned without risk of overheating the tube. The maximum dimensions are 40 in. long × 9 in. diameter. When fitted with the protective cradle the weight is increased from 142 lb. to 171 lb. The control unit is of the carrying case type, the maximum dimensions being 19 in. long × 14 in. wide × 8 in. deep: the weight is 60 lb.

The control operates from a 220 volts 50 cycles single phase supply and accommodates fluctuations of 10% either way. It is fitted with direct reading kilovoltmeter and milliammeter in addition to a synchronous timer which can be arranged for automatic repetition of exposures. The kilovoltage and tube current controls are of the stepless type giving continuously variable control. Among the accessories supplied as standard are a telescopic centre finder (fold back type) end rings for tank head, protective cradle for tank head, 30 ft. mains cable, and 2 × 30 ft. control-to-head cables which can be connected together, thus making a 60 ft. cable if required.

Pantak, Ltd., Vale Road, Windsor, Berks.

Fused Quartz

HANOVIA LAMPS DIVISION of Engelhard Industries, Ltd., Slough, has just taken over the sole agency in the U.K. for the Heraeus Quarzschmelze G.m.b.H., Hanau, West Germany, manufacturers of highest quality fused quartz in both optical and commercial grades for all industrial and laboratory applications.

Heraeus quartz-glass is available in a wide range of sizes as tube or rod, while the optical grades can be furnished as prisms, lenses, blanks, etc. A special homogenised quartz sold under the trade name Homosil meets the highest requirements in the field of optics, since it is without the typical quartz absorption bands at 2,400 AU, and has superior transmission properties in the ultra-violet to pure rock crystal. A wide range of laboratory ware in both transparent and satin silica is offered, and within certain limits pieces can be made accurately to customers' own patterns.

Hanovia Lamps Division, Engelhard Industries Ltd., Slough, Bucks.

Correction

HIGH TEMPERATURE SINTERING FURNACE

WE regret that at the end of the description of the high-temperature sintering furnace on page 89 of the February issue of METALLURGIA, the maker was described as Royal Electric Furnaces of Walton-on-Thames, instead of Royce Electric Furnaces, as appeared in the text.

CURRENT LITERATURE

Book Notices

TRACE TECHNIQUES

USING THE K 1,000 CATHODE RAY POLAROGRAPH

Volume 1, by J. Hetman. 48 pp., Southern Instruments, Ltd., Camberley, Surrey. 25s.

THIS book is a collection of thirty methods, developed by the author, for the determination of trace constituents and impurities in a wide variety of materials. Both inorganic and organic substances have received attention. Many of the methods will be of direct or indirect interest to metal analysts, as they include the simultaneous determination of copper, nickel and cobalt impurities in iron; of copper, lead and zinc in manganese brass; copper and lead in leaded steel; lead in brasses and bronzes; and the simultaneous determination of beryllium, aluminium and lead. A method for the direct determination of traces of cyanide (less than 1 p.p.m.) in water will be of interest to those concerned with effluent problems. Methods for the determination of trace metal impurities in foods and organic materials are also included. A bibliography, giving some background information and references to work of allied interest, is appended.

The methods are in note form giving, in addition to brief details of procedure and interpretation of results, essential information such as preparation of sample, base electrolyte, reference electrode employed and recommended instrument settings. Specimen polarograms are included in each case, but neither theory nor suggestions for extension of the methods to other materials are given. This information would have been of great value and would have added to the value of the work. In addition, the very high sensitivity and low levels of detection which can be achieved by this technique have not been sufficiently emphasised, and have to be inferred from the specimen polarograms and concentrations in the calibration solutions.

The methods are easy to follow and contain sufficient information for direct application to the materials described. As indicated above, however, they suffer from the brevity of the treatment and a number of errors are present. The most persistent of these is the use of "basic" instead of "base" electrolyte, which is an error sometimes encountered in foreign papers written in English. The heading "Basic Electrolyte" is itself sometimes misleading, since the materials listed under it in several methods do not have this function. The use of the term "diffusion current" to describe the current flowing at the peak of the wave is also incorrect, since the current flowing at this point is not diffusion controlled: "peak current" is to be preferred.

The book starts very abruptly with a short Foreword, but without an Introduction. The inclusion of a section setting out objectives and providing a theoretical background would have given the work an enhanced value, especially if details of the advantages of cathode ray polarography had been included. This would have helped non-specialist and potential users to realise that no new technique employing special and exclusive principles is involved, but that this is an advance in polarographic instrumentation offering greatly increased sensitivity and rapidity of operation.

No collection of methods for a single polarographic technique has previously been published in this way and this, therefore, makes a valuable contribution to the literature. In spite of obvious limitations it will be of value, not only to the specialist, but also to polarographers and analytical chemists in general, who will find much of the information useful outside the present field.

G. F. REYNOLDS

METALLIZING HANDBOOK

320 pp., more than 150 diagrams, photographs and charts. Metallizing Equipment Co., Ltd., Chobham, Woking, Surrey. 35s. 10d. (post paid in the U.K.).

A NEW edition of the Metallizing Handbook has just been published, covering techniques and developments in flame spraying. Like its predecessors, it is written for owners and operators of metallizing equipment, and for design, production and maintenance engineers. The book is in two parts covering standard practices and developments in metallizing by flame spraying. Part one is devoted to wire metal spraying and part two contains the latest information on the powder process. Metal spraying and ceramic spraying are each dealt with in great detail, and a section is devoted to the latest techniques in the application of hard-facing materials, including tungsten carbide. The text gives practical and technical information on preparation, spraying and finishing, while data is included to assist in the estimation of costs.

Trade Publications

METALECTRIC FURNACES, LTD., have recently issued a reprint from the *Institute of Vitreous Enamellers Bulletin* entitled "Electric Furnaces for Fusing." It is the author's belief that insufficient knowledge of the construction and running of the modern electric furnace has caused some of its advantages to be overlooked, and in this article he proceeds to rectify the situation by describing the types of furnaces available for this class of work.

ILLUSTRATED by pictures in colour and in black and white, and by perspective drawings with the gas distribution shown in colour, a brochure recently produced by Woodall-Duckham Construction Co., Ltd., describes the Murton coking plant of the Durham Division of the National Coal Board. This is a reprint from "*Coke and Gas*," with additional illustrations and a description of the oven heating system. The carbonising capacity of the plant is 1,000 tons of coal a day, from which some 250,000 tons of high grade metallurgical coke will be produced annually. Purified gas for the gas grid will be available at the rate of 7¼ million cubic feet a day and the by-product plant will produce concentrated ammoniacal liquor, tar, and about one million gallons of crude benzole annually. An outstanding feature of the plant is its advanced system of centralised instrumentation to assist operational control.

For some years Johnson, Matthey & Co., Ltd., have produced the rare earths, and recent research into the application of ion-exchange techniques to the separation of these materials has made them available in large quantities, in various grades of purity, and at much lower cost than hitherto. New techniques have also been applied to the production of the rare earth metals and all the fourteen " lanthanons " that occur naturally and the related elements scandium and yttrium are now available in a state of high purity. All of the sixteen metals have been remelted into ingots or rods. Lanthanum, cerium, neodymium, praseodymium, yttrium and gadolinium have been successfully extruded, and subsequently drawn to fine wire. A publication, " Products of the Rare Earth Group," just issued by the company, describes the properties, characteristics and availability of this potentially valuable and very topical range of materials. This is available free on request to the company's head office at 73-83 Hatton Garden, London, E.C.1.

Abstracts of literature in nickel-containing heat- and corrosion-resisting materials form an interesting feature of the March issue of *The Nickel Bulletin*. Ranging in subject matter from the high-temperature properties of, and phase reactions in, heat-resisting alloys, to residual-oil-ash corrosion, metal dusting and the resistance of alloys and steels to attack by various corrosive media, they provide an apt illustration of the important role played by nickel-containing materials in this field. Worthy of note are references to papers reporting work on the evaluation of aluminium-nickel alloys in relation to nuclear-power applications. The other sections of the issue contain items on, *inter alia*, the determination, properties and uses of nickel, the corrosion-resistance of chemically deposited nickel coatings, the properties of nickel-containing gunmetals, welding and hardenability of cast irons, and the properties of low-alloy steels.

For the purpose of the new information publication entitled " Applications of Molybdenised Lubricants in the Iron and Steel Industry," of Rocol, Ltd., Rocol House, Swillington, near Leeds, the term " Iron and Steel " is considered to apply to the heavy industrial processes of the production of steel stock, forgings and heavy castings from the preparation of the ore down to finished wire rod, bar and structural shapes ; castings ; hot rolled plate ; hot and cold rolled sheet and strip ; drop, heavy and light forgings ; and tubes. The publication gives a comprehensive list of the applications of the lubricants subdivided into main processes, with all similar equipment grouped together.

John Summers & Sons, Ltd., one of Britain's largest sheet steel manufacturers, have just published the first edition of their new *John Summers Review*. Issued quarterly, this four-page publication with colour front and back pages, will regularly provide metal manufacturers, industrial designers, architects and other technicians with up-to-date details of applications of sheet steel, particularly John Summers' plastic-coated steel, Stelvetite. This first issue describes Luxfer partitions, Blend occasional furniture, Smiths clocks, the Triumph TR3 sportscar facia panel, and Summerscales Double-Spin combined washing-machine and spin-dryer—all finished in Stelvetite. Copies of the *John Summers Review*, produced jointly by the Company's advertising agents and public relations consultants, the Basil Butler Co., Ltd., and Hurd Public Relations, Ltd., are being distributed to interested concerns in Britain and overseas. Further copies may be obtained direct from John Summers & Sons, Ltd., Hawarden Bridge Steelworks, Shotton, Chester.

Five new single sheet leaflets, Nos. IN.1, IN.3, IN.4, IN.5 and IN.6, give details of Incanite—the special process cast iron made by The Incandescent Heat Co., Ltd. These have been designed to show a few of the many applications of this material, which include machine bases, slides, gears and rollers ; pressure tight housings and cylinders ; press tools ; drawing dies ; and cams and gears.

An interesting application of Bonderizing, namely the production of an oxalate coating on stainless steel wire stock prior to cold heading in a bolt making plant, is described in the April-May 1960 issue of *The Bonderizer*. This treatment was successful in overcoming the difficulties encountered due to scoring and galling, which tend to occur when stainless steel comes into contact with the tool surface. This issue of *The Bonderizer* is devoted exclusively to cold forming, and other items deal with the deep drawing of instrument cases, cold extrusion of steel at the R.O.F., Birtley, shaped bar drawing, seamless steel tube production, and steel wire production, in all of which Bonderizing is used to facilitate the cold forming operation.

As a result of considerable publicity in the form of papers and discussions on the subject of centrispun rings, the engineer has a fair knowledge of the potential uses of the standard centrispinning process. In a leaflet, Publication No. 518, on centrifugally spun boiler tube plates and similar heavy section discs, Firth-Vickers Stainless Steels, Ltd., aim at convincing the engineer that it is possible to spin discs without the usual hole in the centre, and with adequate soundness for use under onerous working conditions. Results are given of tests taken on a centrifugally cast tube plate showing that the material is sound at the centre.

" Wiggin High-Nickel Alloys v. Sulphuric Acid " is a new publication issued by Henry Wiggin & Co., Ltd. It is the latest of a series of publications issued by the company, dealing with the corrosion-resisting properties of its high-nickel alloys. These are well-known in the field of chemical and process engineering and this publication, invaluable to the engineer and designer, gives highly detailed information, with many photographs and tables, on their particular properties. Monel, "AT " nickel, Inconel, Ni-o-nel and the Corronel alloys are particularly featured in well proven applications. Examples of their successful uses in pickling equipment, chemical plant, food processing equipment and petrochemical plant are featured.

Books Received

" Aluminium Busbar," By A. G. Thomas and P. J. H. Rata. 105 pp. inc. index. London, 1960. Hutchinson & Co. (Publishers), Ltd., for Northern Aluminium Co., Ltd. 21s. net.

" Nature and Properties of Engineering Materials." By Z. D. Jastrzebski. 571 pp. inc. index. New York and London, 1959. John Wiley & Sons, Inc., and Chapman & Hall, Ltd. 88s. net.

" The Instrument Manual." Third Edition. 742 pp. inc. index. London, 1960. United Trade Press, Ltd. 105s.

LABORATORY METHODS

MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC

INSTRUMENTS AND MATERIALS

MAY, 1960 Vol. LXI, No. 367

Portable Creep-Testing Unit with Autographic Extensometer

By C. Wheatley*, M.Sc., G.I.Mech.E., and R. Grosvenor†

A creep testing machine is described which is sufficiently versatile to meet the majority of conditions currently needed or likely to be required in the foreseeable future for both creep and stress-rupture tests. The machine is capable of testing specimens over the load range 0·01–1 ton, and the extensometer records creep extensions autographically. Furnace temperatures up to 1,050° C. can be controlled at ±1° C. by an electronic temperature control system which eliminates the risk of failure arising from the sticking of the electro-mechanical relays used in the various modifications of the "Prosser" system.

LABORATORIES engaged in the testing of high-temperature alloys employ, among other equipment, stress-rupture and high-accuracy creep-testing machines of various types, each designed to meet a particular range of test conditions. At the Research Laboratory of The Mond Nickel Co., Ltd., 200 stress-rupture and 50 high-accuracy creep-testing machines of five different types have been in service for several years, and the experience gained in operating them indicated that it would be possible to design a new machine of more "universal" type, sufficiently versatile to meet the majority of testing conditions which are currently needed or likely to be required in the foreseeable future.

The following specification for such a machine was drawn up as a basis for design :—

(1) The machine should be self-contained, with the exception of temperature-measuring equipment, and should meet the requirements of British Standard 1686 : 1950—Long-period, high-sensitivity, tensile creep testing.

(2) The machine should test specimens over the load range 0·01–1 ton.

(3) The furnace temperature and control equipment should be capable of providing and regulating specimen temperatures up to 1,050° C., controlled to ±1° C.

(4) The extensometer should be capable of autographically recording creep extension up to 0·10 in.

(5) Apparatus for recording the time to fracture of the specimens should be incorporated in the machine.

The machine would incorporate a rigid frame in which various types of furnace, test specimen, extensometer and equipment for controlling furnace atmospheres could be fitted : it should be equipped with a counter-balanced loading lever and should have self-aligning seatings in the specimen-shackle assembly, to assist axial loading of the specimen.

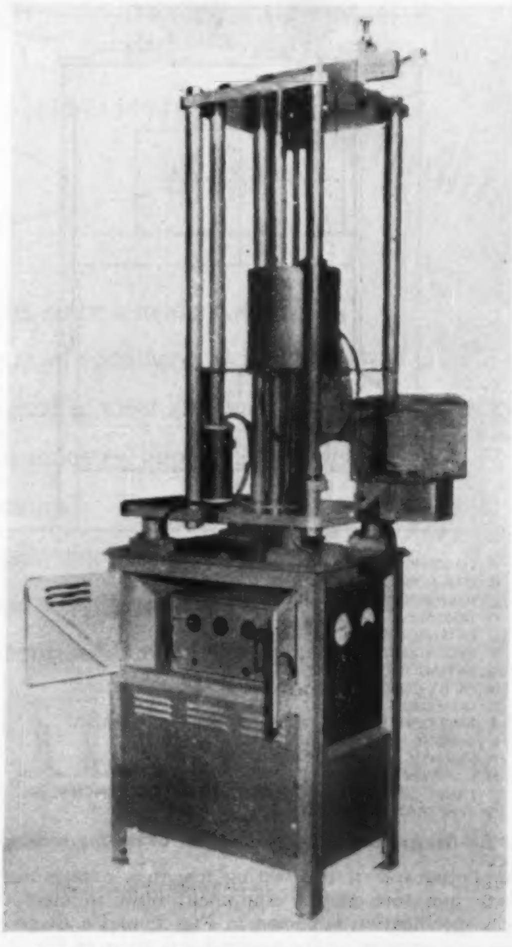
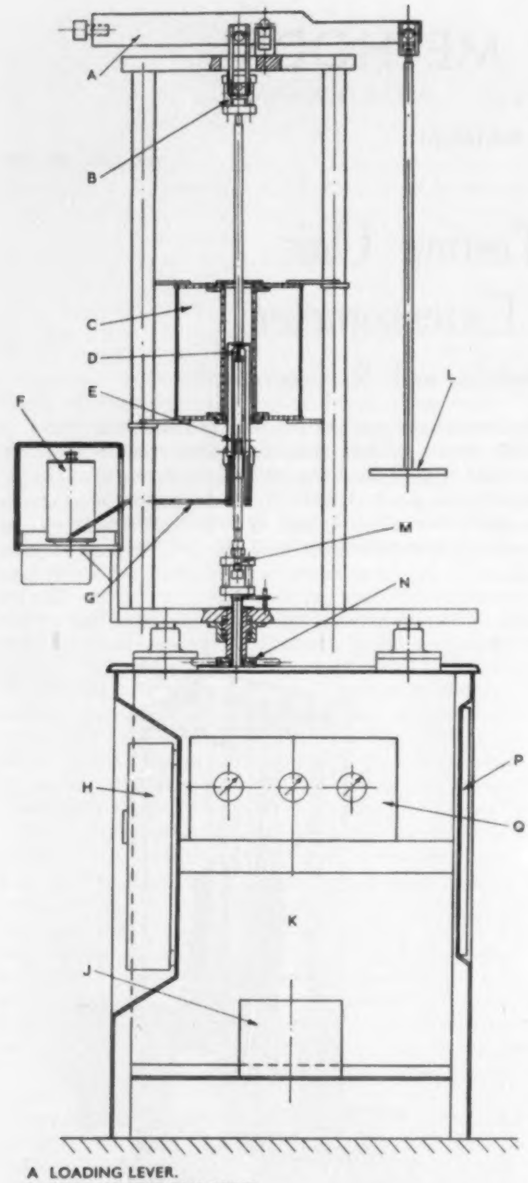


Fig. 1.—The complete testing machine.

* The Mond Nickel Co., Ltd.

† The Electronic and Mechanical Engineering Co., Ltd.



A LOADING LEVER.
B SELF-ALIGNING BALL JOINT.
C FURNACE.
D SPECIMEN.
E EXTENSOMETER.
F RECORDING DRUM.
G EXTENSOMETER LEVER.
H PANEL CONTAINING FURNACE GRADIENT RESISTORS.
J SATURABLE REACTOR.
K BASE CONTAINING ALL THE ELECTRICAL APPARATUS.
L WEIGHT PAN.
M SELF-ALIGNING BALL JOINT.
N CAPSTAN SCREW.
P PANEL CONTAINING ALL SWITCHES, CLOCK, AMMETER, etc.
Q TEMPERATURE CONTROLLER.

Fig. 2.—Diagrammatic arrangement of testing machine.

A photograph of the testing machine, extensometer and temperature-control equipment built to meet the above specification is shown in Fig. 1, and a diagrammatic arrangement of the apparatus is given in Fig. 2.

Creep-Testing Machine

The machine consists essentially of a four-pillar rigid framework, mounted, through shock absorbers, onto a cabinet containing the electronic and electrical equipment necessary for the control of the temperature of the specimen. The loading lever mounted on the top plate of the machine incorporates a movable knife edge which can be rotated, and a counterbalance weight which extends the lower value of the loading range almost to zero. During calibration the movable knife edge in the loading lever is adjusted and locked to give a lever ratio of 11·2. To assist axial loading of the specimen, self-aligning ball seatings are fitted to each of the shackles which hold the specimen. Attached to the bottom ball seating, a capstan screw under the bottom plate enables the loading lever to be re-set when excessive creep strain occurring in the specimen might cause the loading lever to foul the top plate of the machine. During re-setting of the loading lever no torque is transmitted to the specimen, as rotation of the shackle assembly is prevented by a rigid torque arm.

The counterbalanced electric furnace is maintained continuously at test temperature, and is guided between the pillars of the machine, being raised for replacement of fractured specimens. Temperature of the specimen is measured by means of one platinum / 10% rhodium-platinum thermocouple strapped to the centre of its gauge length.

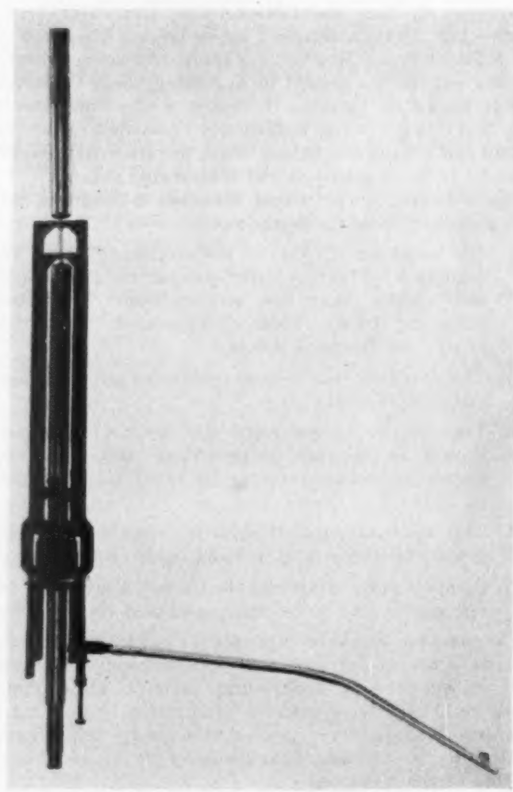


Fig. 3.—Autographic strain recording extensometer.

Extensometer

The autographic strain-recording extensometer designed for use with this equipment is shown in Fig. 3. It consists of two pairs of comparator legs (one pair from each side of the specimen), held onto the location ridges by springs and link plates outside the furnace, and stabilised against the lower shackle by roller pivots. The relative movement between the legs of one pair is mechanically magnified ($\times 50$) by a light lever, the fulcrum of which is a steel pin accurately located through reamed holes in the lever and the inner leg of the extensometer : the second pivot is another steel pin fixed into the lever and resting on the brass re-setting slide carried in the outer leg. To the outer end of the lever is attached a recorder pen, which traces out the creep-strain curve on a chart carried by a rotating drum. The inner leg of the extensometer is mounted upon the lower location ridge of the specimen, below which only negligible creep strain occurs : there is consequently no vertical movement of the lever fulcrum relative to the recording drum. This arrangement ensures a fixed baseline to the autographic record. The recording drum has a two-speed gear box giving two chart speeds, 1 in. in 2 hours and 1 in. in 20 hours. This extensometer can be fitted to specimens having gauge lengths of 0·5–1·5 in. and diameters of 0·083–0·250 in.

A similar extensometer, fitted with a differential transformer-type transducer between each pair of comparator legs, replacing the single lever, has also been developed. This equipment is capable of giving an autographic record either of the separate strains on each side of the specimen or of the mean value of these two separate strains. The extensometer has a linear range of 0·1 in., with a chart sensitivity of 5×10^{-5} in. at an amplification of $\times 600$.

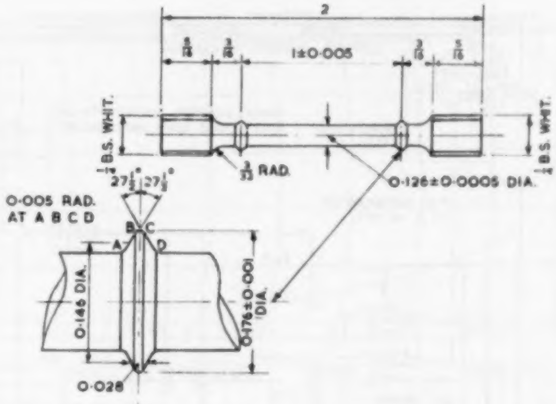


Fig. 4.—Miniature ridged specimen (all dimensions in inches).

Test Specimen for Autographic Extensometer

The form of test specimen was selected to give the greatest possible accuracy in the strain measurements, and in conjunction with the extensometer to induce, into the specimen, the minimum possible force other than the test load. The specimen used is shown in Fig. 4, where

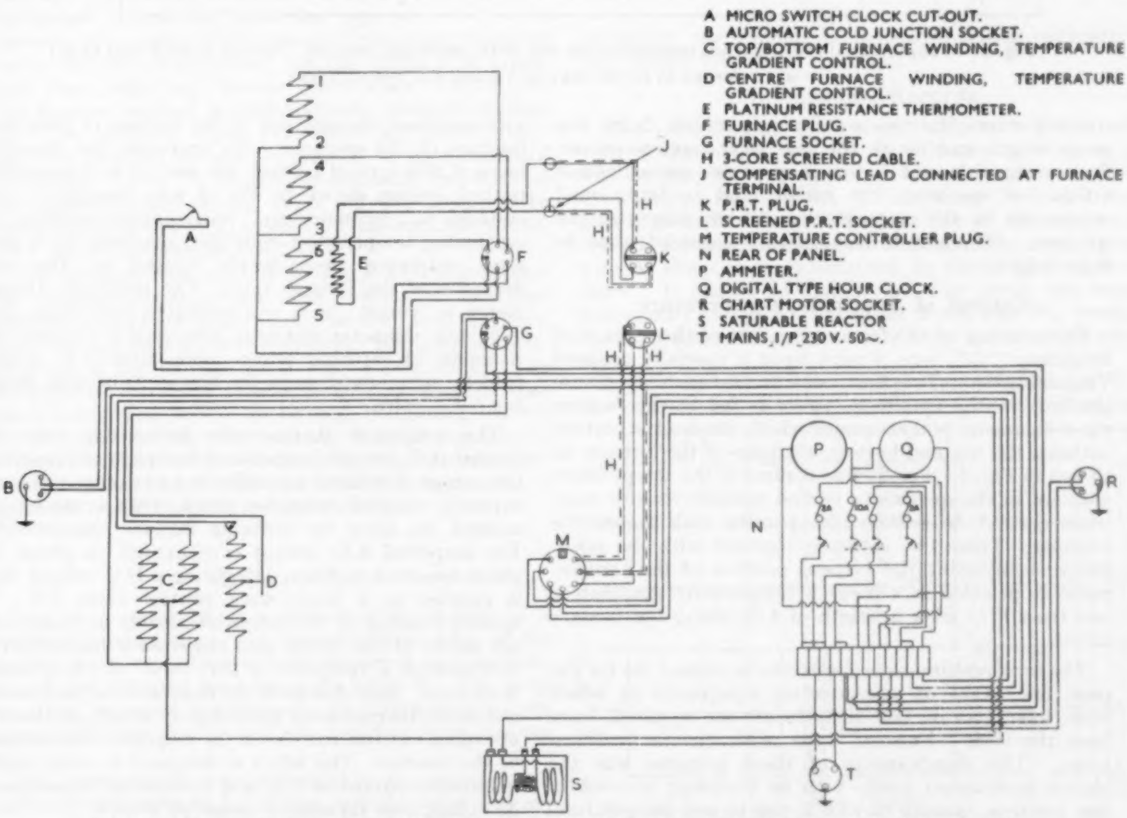


Fig. 5.—Circuit diagram of creep testing unit (sockets viewed from rear, plugs from front).

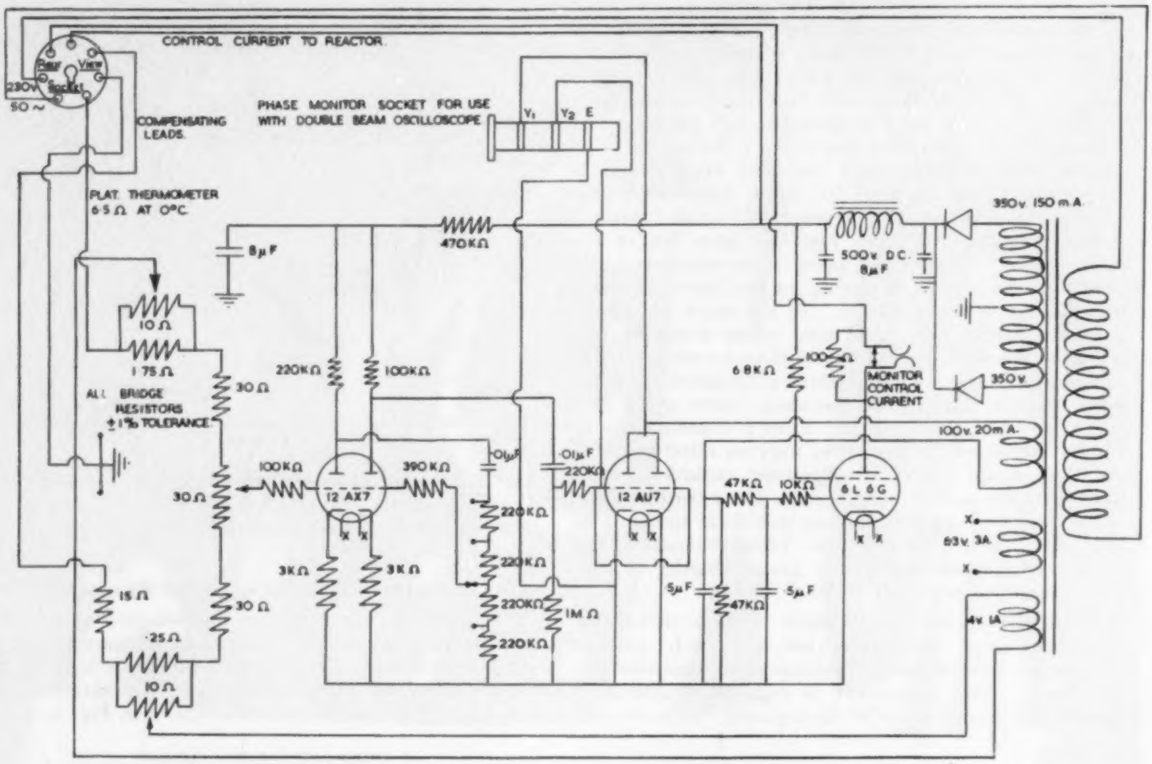


Fig. 6.—Proportional temperature controller for use with saturable reactor. Valves 12AU7 and 6L6G are referred to in the text as V2 and V3, respectively.

a detail shows the two small ridges which define the gauge length and locate the legs of the extensometer. These ridges do not induce any stress concentration within the specimen, but rather tend to form small constraints on the material close to the ridges on the specimen. In no test has fracture occurred close to these ridges.

Control of Specimen Temperature

The winding of the furnace consists of three zones of Brightray " C " wire, wound upon a specially grooved Vitreosil tube. To effect control of the temperature gradient on the specimen, power is fed to the furnace via a balancing potentiometer which divides the current between the top and bottom windings of the furnace, as shown in Fig. 5. For further control of the temperature gradient in the specimen a second variable resistor may, when desired, be switched in, parallel with the middle winding. These two controls, together with the possibility of adjusting the vertical position of the furnace, make it possible to achieve a temperature gradient of less than 1° C. over a length of 4 in. along the furnace tube.

The temperature-control systems in normal use for the past few years on creep-testing equipment in which close tolerances on test temperature are required, have been the basic " Prosser " type, with various modifications. The disadvantage of these systems was the electro-mechanical relay, with its tendency to stick in one position, causing inevitable rise in test temperature, with resultant burning out of the furnace or premature fracture of the specimen. To overcome the disadvantages of this type of control, the electronic temperature-control system shown in Fig. 6 was developed. This employs a " contactorless," continuously-variable, proportioning temperature-controller, operated by a platinum resistance thermometer located on the outer diameter of the furnace tube. The resistance thermometer is wound, in a non-inductive mat form, from 0·25 mm. diameter platinum wire, and is enclosed in a Nimonic 75 earthing screen made from 5 in. wide × 0·01 in. strip bound on to the furnace tube with Brightray " C " wire.

The resistance thermometer forms one arm of a normal A.C. 50-cycle impedance bridge in the controller, the output of which is amplified in a two-stage resistance-capacity coupled amplifier fitted with a pre-set gain control, to allow for differing furnace characteristics. The amplified A.C. output is compared, in phase, in a phase-sensitive rectifier, *V*2, giving a D.C. output which is applied to a heavy-duty control valve *V*3. The control winding of the saturable reactor is connected in the anode of this valve, and the reactor-control current is therefore a function of the value of the platinum resistance. Any change in the resistance of the platinum resistance thermometer immediately results in alteration of control current and also of the magnetic characteristics of the reactor. The latter is designed to work up to a maximum current of 8 A. and to have an impedance, at 50 cycles, over its control range, of 10 to 1.

Outside the limits of $\pm 10°$ C. of test-piece temperature, the reactor has maximum or nil input to its control winding, resulting in maximum or minimum power input to the furnace. Between these limits the furnace current will take up some value sufficient to maintain the required resistive value of the resistance thermometer. The saturable-reactor system will control the furnace on this machine over the range 150°–1,120° C. Higher temperatures have not been attempted, since the maximum working temperature for a reasonable furnace life is 1,050° C. The control characteristics of this system have been compared directly with a " Prosser "-type temperature-controller, using an electronic recorder capable of recording to 0·04° C., using a platinum/10% rhodium-platinum thermocouple. There was no detectable difference between the two systems : each resulted in " straight-line control."

The main advantages of the new control system are its inherent reliability, the possibility of dispensing with electro-mechanical relays and their tendency to stick in one position, and its ease of operation, since the only adjustment required is that of the control-bridge resistors. These resistors are arbitrarily calibrated to suit the particular type of furnace. The only possible causes of failure are the valves in the electronic equipment, but the circuit is so arranged that with any failure other than that of *V2* the furnace current is automatically reduced. Failure of *V2* is unlikely, since this valve is " under run " and two sections are used in parallel, to give increased reliability. After 18 months' continuous operation of one controller the control valve *V3* was found to be operating at about 30% efficiency ; the controller, however, was still functioning correctly.

The machine-base/control cabinet is so designed that the temperature-controller and associated electrical instruments are not unduly influenced by heat radiation from the gradient-control resistors : the internal temperature of the cabinet never rises above 45° C. The temperature-controller cabinet is also designed so that there is a natural flow of air through ventilating louvres at the bottom of the case and out through top louvres adjacent to the control valve and the selenium rectifiers. These features ensure that the electrical equipment is operated under reasonable conditions.

Calibration

The creep machine was calibrated by means of a certified proving ring. The movable knife edge of the loading lever was adjusted until the lever was accurate to $\pm 0{\cdot}5\%$ over the load range 22–2,240 lb. The lever was then balanced, weighing the counterbalance weight against the top shackle and the top two extensometer legs.

The extensometer was calibrated in an extensometer calibrator sensitive to 1×10^{-5} in. and the lever was set to give a magnification $\times 50 \pm 0{\cdot}5\%$. Very little

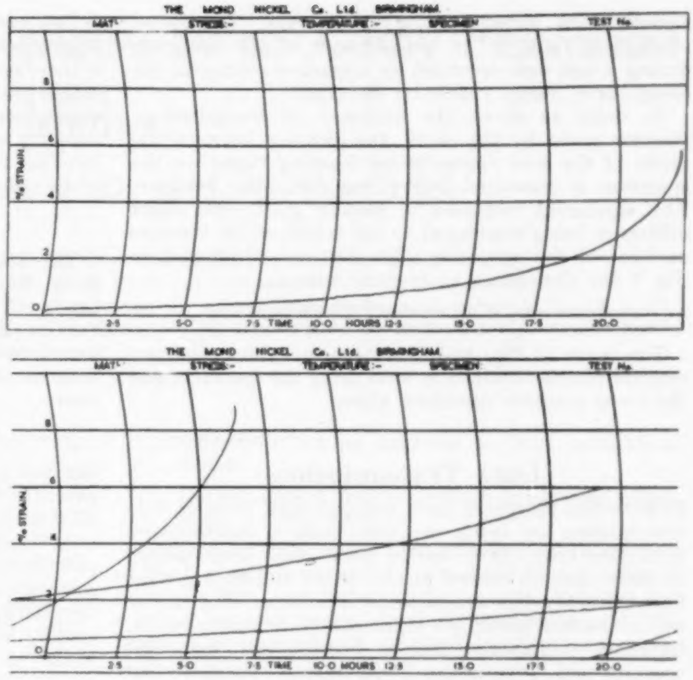


Fig. 7.—Autographic creep strain records.

hysteresis was evident in the extensometer-calibration line, which was linear in form.

Autographic Records

Two typical autographic creep-strain curves, obtained with a chart speed of 1 in. in 2 hours, are shown in Fig. 7 : they show creep curves for specimens with lives to fracture, respectively, of less than 24 hours and greater than 24 hours. When the life of the specimen extends for more than one revolution of the chart drum a spiral creep curve is obtained, as shown in the lower chart. It should be noted that this curve has been interrupted at approximately 67 hours and 6% strain, at which stage the extensometer lever was re-set to zero strain.

In all tests with the temperature controlled to $\pm 1°$ C., the extensometer has recorded a creep curve which is

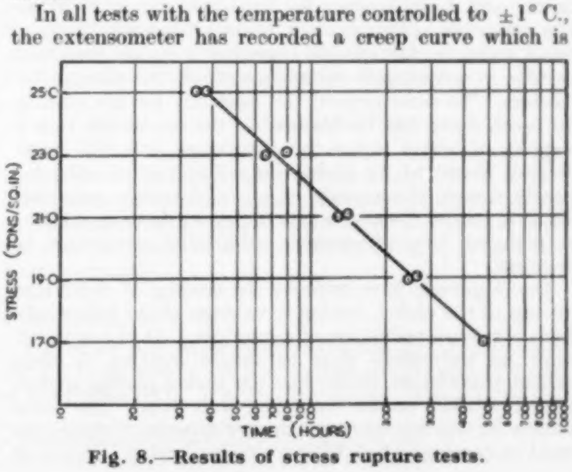


Fig. 8.—Results of stress rupture tests.

smooth from initial loading to fracture. Controlled changes of $\pm 10°$ C. in temperature of the specimen during a test were recorded by a marked change in the creep curve, easily visible on the chart.

In order to check the accuracy of elongation-at-fracture given by the chart, the distance between the crests of the two extensometer locating ridges on the specimen is measured before test and after fracture. The agreement obtained is usually good, the slight difference being attributed to the misfit of the fracture surfaces of the specimen. In the tests illustrated in Fig. 7 the elongations at fracture were :—

Chart elongation 5·6% : measured elongation 6·2%
Chart elongation 14·8% : measured elongation 15·2%

The curve in Fig. 8 shows a typical series of stress-rupture results obtained in tests using the specimen and the creep machine described above.

This creep-testing machine has satisfactorily met the original specification, and for the past two years twelve of these machines have been in service, with no operating difficulties. The experience gained has shown that the design could be further improved by increasing the capacity to 2 tons and incorporating automatic beam levelling for use on specimens which exhibit abnormal creep strain.

Acknowledgments

The authors acknowledge their indebtedness to The Mond Nickel Co., Ltd., for permission to publish this paper. They also wish to thank the National Gas Turbine Establishment (Plastic Flow Department) for permission to use, as a basis, a design from which has been developed the autographic extensometer described above.

Data Transmission

ELECTRONIC computer users and potential users of data transmission are being circulated with a questionnaire from the Post Office asking what data transmission facilities may be needed in the future and saying what Post Office services are currently available. The survey will be made of about 350 organisations including banks, insurance companies, finance houses, and industrial concerns and Government Departments. As more and more computer and punched card equipment is brought into use for commercial, industrial and scientific purposes the necessity for new and sometimes more speedy transmission of data arises. This usually means sending information from branches or departments of an organisation to a central point for processing computer or punched card equipment and sometimes the return of information to the originating point after processing. The Post Office is aware that it will have a part to play in this new field. It already has a varying range of apparatus and lines which can be used for this purpose and is anxious to keep abreast of events and to develop new facilities.

Better Chromium Plating

THE deterioration of plating, particularly on motor cars, has caused a certain amount of criticism in recent years. Many of the components, door handles, lamp bezels and boot hinges, for example, are made from zinc alloy die castings, and these plated die castings have come in for special comment because corrosion results in permanent disfigurement of the plating by blisters. To some extent, the tendency for the plating to break down can be blamed on the use of too thin a deposit of nickel under the chromium, and the latest British Standard for nickel-chromium plating calls for nearly double the amount of nickel formerly specified. Even so, under the severe test of day by day exposure to a polluted city atmosphere additional protection is required.

One approach is to improve the coating of chromium on top of the nickel, taking advantage of the inherently high corrosion resistance of chromium. At the moment only an extremely thin chromium coating is used (about 0·00001 in. thick) and the nickel plating underneath is relied on for corrosion protection. The main reason for this has been that thicker deposits of chromium tend to crack and lose their protective value. Research has now shown how to overcome this difficulty, at least partially, to produce coatings two or three times as thick without cracks developing.

A booklet, "Better Plating on Die Castings," just published by The British Non-Ferrous Metals Research Association shows that the extra corrosion resistance of the plating is quite remarkable, even though the chromium coating is still well below 0·0001 in. thick. While it would be wrong to claim indefinite life for this new chromium plating, the use of which is not confined to die castings, its adoption should result in much better serviceability from plated components. "Better Plating on Die Castings" discusses the modifications to plating techniques required for applying the improved coating and the control tests needed to ensure that the best results are obtained. It is available from The British Non-Ferrous Metals Research Association, Euston Street, London, N.W.1., price 7/6d.

E.S.C. Tinsley Park Scheme

ENGLISH STEEL CORPORATION, LTD., announce that the consent of the Iron and Steel Board has been obtained to further developments at Tinsley Park, Sheffield. It is proposed to replace billet mills installed at River Don and Stevenson Road works more than forty years ago with a modern installation, comprising one 42 in. blooming mill and two 32 in. billet mills, which will permit more economical production of a larger volume of alloy and special carbon steel billets. The capacity for billet production will be increased by 250,000 tons per annum, some of which will be processed in other plants of the group. In addition, the bar rolling mills at River Don works are being replaced by new mills which will give an anticipated additional output of 60,000 tons per annum.

To provide the additional steel required, three 100-ton electric melting furnaces are being installed, and with this addition the total melting capacity of the E.S.C. group for the production of alloy and special carbon steels will be approaching 1,000,000 tons per annum. The estimated cost of the project is £26 million and it is planned to be in full operation in 1963. The plant will be operated by English Steel Rolling Mills Corporation, Ltd., a wholly-owned subsidiary company. The proposals for a heavy forge at Tinsley Park which were approved in 1956 have been deferred.

Analysis of Extracted Residues by an X-Ray Fluorescent Method*

By H. Hughes, B.Sc., Ph.D., and L. Gwilliam, B.Sc.†

A method has been developed for the X-ray fluorescent analysis of small amounts (0·1 g.) of residues extracted electrolytically from alloy steels. A comparison between analyses by this method and analyses of the same specimens by conventional methods shows good agreement, and further applications of the technique are described. The method is not limited to extracted residues, but may be used on any material in powder form, e.g. oxide scales, corrosion products, etc.

OVER the last few years great progress has been made in the study of the metallurgy of alloy steels. X-ray diffraction techniques have been available for some time to identify the various phases precipitated in alloy steels, and the electron microscope is now used extensively both for microscopy and diffraction work. These techniques give information about the phases present and their mode of occurrence. The actual composition of a phase, however, may vary considerably (for example $M_{23}C_6$), and if the composition of a residue could be ascertained, such information, together with that already being obtained from established electron microscope and X-ray techniques, would give a clearer picture and a better understanding of the precipitation processes taking place in alloy steels. For reasonably accurate chemical analysis a sufficiently large amount of residue (0·5–1·0 g.) is required. In most cases it is a long and tedious process to obtain this amount and the actual analysis can also be time-consuming.

The development of the fluorescent spectrometer has opened up new possibilities. The last few years have seen considerable progress in the field of X-ray fluorescent spectroscopy. This progress is illustrated, for example, by numerous and diverse papers in the proceedings of the 4th,[1] 5th,[2] 6th[3] and 7th[4] Annual X-ray Conferences on the Industrial Applications of X-ray Analysis at the Denver Research Institute, University of Denver. Of special interest is a paper by F. Claisse[5] and another by Houk and Silverman[6] which was presented at the 5th Denver Conference.

The present paper describes a method which has been developed with the above special application in mind.

General Considerations

The greatest difficulties to be overcome in X-ray fluorescent spectroscopy are associated with inter-element effects, i.e. the effect of a variable amount of a second element on the counting rate for the element being analysed. This has been overcome by dilution of the specimen to be analysed with a comparatively large amount of diluent by fusion in [5] and by solution in [6] the latter. Claisse[5] discusses this point in some detail.

Neither of these two dilution methods, as it stands, satisfies all the requirements for extracted residues. Thus :

(1) A fusion method in itself is not feasible because of the difficulty of obtaining carbide residues of sufficiently varied analyses to give calibration curves.

(2) The solution of carbide residues in acid can sometimes be very difficult.

A compromise between the two methods would appear to be the best approach, and a method has been developed on these lines, i.e. fusion of the residue, followed by solution in acid under standard conditions and the preparation of synthetic standard solutions under the same conditions.

Experimental Details

Preparation of Residues in Solutions

0·1 g. in 25 ml. of solution was considered to be a reasonably small amount of residue with which to work. It is possible, however, to start with less and finish up with a proportionally smaller volume of solution (12 ml. is, in fact, sufficient with our present specimen holder to give infinite depth, and one could start with 0·05 g. of specimen and make up to 12·5 ml. : even less would be required with a specimen holder of smaller cross-sectional area). This would be expected to give a less accurate result.

Experiment showed that the best fusion mixture was

0·1 g. of sample
0·3 g. of fused borax
0·8 g. of sodium carbonate.

This was fused at approximately 1,100° C. and the mixture then dissolved in 25 ml. of nitric acid (sp. gr. 1·20), the solution being warmed. If necessary, a few drops of nitric acid (sp. gr. 1·20) were added to the solution in a 25 ml. measuring flask to allow for evaporation.

Preparation of Standard Solutions

For direct comparison, the concentration of the elements in the standard solutions should be 4 g. per litre and they must also contain the correct concentrations of Na, B, O and N as the solution to be analysed to give the same counting conditions (the effect of hydrogen has been neglected).

Iron, Cobalt and Nickel

4 g. of Fe (Co, Ni) is dissolved in 500 ml. of nitric acid (sp. gr. 1·20) and 51·33 g. of sodium nitrate and 22·75 g. of borax ($Na_2B_4O_7 . 10\ H_2O$) then added. The NO_3 in the

* A communication from The United Steel Companies Limited.

† Mr. L. Gwilliam is now with The Steel Company of Wales Ltd.

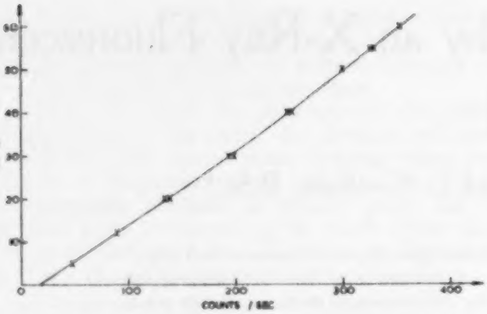


Fig. 1.—Calibration curve for iron (using slit 2).

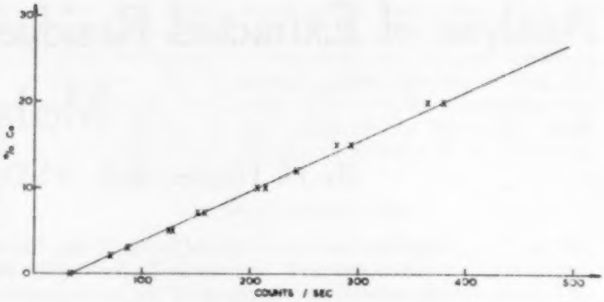


Fig. 2.—Calibration curve for cobalt.

sodium nitrate is equivalent to that in 100 ml. of nitric acid (sp. gr. 1·20), and the solution is made up to 1 litre with 400 ml. of nitric acid (sp. gr. 1·20) and 100 ml. of water. Standard solutions of Cu, Zn and Mn may be prepared in the same way.

Chromium

9·6 g. of ammonium dichromate ($(NH_4)_2Cr_2O_7$) is dissolved in 100 ml. of water. 400 ml. of nitric acid (sp. gr. 1·20) is added, and then 51·33 g. of sodium nitrate and 22·75 g. of borax ($Na_2B_4O_7$. 10 H_2O). The solution is made up to a litre with a further 500 ml. nitric acid (sp. gr. 1·20).

Vanadium

9·18 g. of ammonium meta-vanadate (NH_4VO_3) is dissolved in 500 ml. of nitric acid (sp. gr. 1·20) and 51·33 g. of sodium nitrate and 22·75 g. of borax ($Na_2B_4O_7$. 10 H_2O) added. The solution is made up to 1 litre with a further 400 ml. of nitric acid (sp. gr. 1·20) and 100 ml. of water.

Molybdenum

6·0 g. of molybdic oxide (MoO_3) is dissolved in 200 ml. of ammonia solution (50% v/v), 300 ml. of nitric acid (sp. gr. 1·20) is then added, followed by the fixed amounts of sodium nitrate and borax. The solution is made up to a litre with a further 500 ml. of nitric acid (sp. gr. 1·20).

Titanium

Titanium presents some difficulty. 4 g. of titanium is dissolved in sulphuric acid (25% v/v) and after solution $Ti(OH)_2$ is precipitated by neutralising the solution with ammonia. After filtration and washing, the precipitate is dissolved in 500 ml. of nitric acid (sp. gr. 1·20) and the fixed amounts of sodium nitrate and borax added. It is usually necessary to filter this solution before making up to 1 litre with a further 400 ml. of nitric acid (sp. gr. 1·20) and 100 ml. of water. This solution must then be standardised and should be freshly made before use, owing to the possibility of a certain amount of hydrolysis. (In the present work the solution used was, in fact, 3·80 g. of titanium per litre and a suitable correction was made on the titanium calibration curve.)

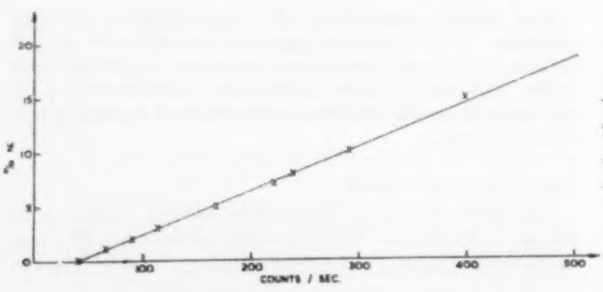


Fig. 3.—Calibration curve for nickel.

25 ml. of a standard of any composition can then be

TABLE I.—ANALYSES OF FOUR 12% Cr STEELS WITH VARYING Ni, Mo AND Co

Steel No.	C%	Mn%	Cr%	Ni%	Mo%	Co%
1	0·103	0·66	11·98	2·09	8·15	10·50
2	0·10	0·59	12·16	2·06	6·30	10·50
3	0·10	0·57	12·19	—	8·90	15·20
4	0·11	0·56	11·99	2·05	6·10	5·12

TABLE II.—ANALYSES OF RESIDUES FROM 12% Cr STEELS WITH VARYING Ni, Mo AND Co

Steel No.	Method	Cr%	Mo%	Co%	Ni%	Fe%
1	X-ray (a)	13·3	28·8	5·2	0·9	45·0
	X-ray (b)	12·9	31·0	5·9	0·5	42·7
	Chemical	13·4	30·4	5·6	0·3	46·1
2	X-ray	13·9	24·7	6·0	0·6	43·7
	Chemical	14·4	24·0	6·1	0·9	48·0
3	X-ray	13·2	29·9	8·9	0·3	38·5
	Chemical	13·4	29·0	9·0	Trace	44·7
4	X-ray	15·9	23·6	3·4	1·6	52·2
	Chemical	15·3	21·0	2·9	1·5	53·6

TABLE IIIa.—COMPOSITIONS OF SYNTHETICALLY PREPARED STANDARD SOLUTIONS CONTAINING Cr, Fe, Co, Ni AND Mo

Solution	Cr%	Fe%	Co%	Ni%	Mo%
1	8	40	7	5	40
2	16	50	2	2	30
3	12	60	5	3	20
4	20	55	10	1	14
5	30	30	20	10	10
6	45	20	15	15	5
7	35	5	3	7	50
8	60	12	12	8	8

TABLE IIIb.—COMPOSITIONS OF SYNTHETICALLY PREPARED STANDARD SOLUTIONS CONTAINING V, Cr, Fe, Co AND Mo

Solution	V%	Cr%	Fe%	Co%	Mo%
1	5	8	40	7	40
2	1	20	55	10	14
3	10	30	30	20	10
4	3	12	60	5	20
5	15	45	20	15	5

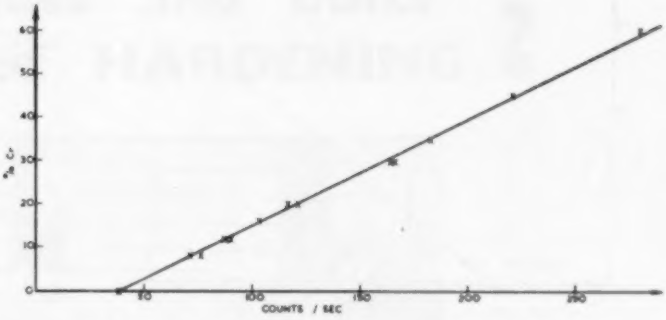


Fig. 4.—Calibration curve for chromium.

made up by mixing the correct proportions of the various synthetically prepared solutions.

Analysis of Residues of Known Chemical Analysis

Analysis of Residues from 12% Cr Steels with Varying Ni, Mo and Co.

Residues obtained from specimens of four casts (whose analysis are shown in Table I) heat treated for 24 hours at 850° C. had previously been chemically analysed. The figures for Fe, Co, Ni, Cr and Mo are shown in Table II.

A sample of 0·1 g. of each residue was prepared for examination by the method already described. (X-ray photographs of the residues had previously shown each to be a mixture of *Chi* phase and M_6C in approximately equal proportions.) Eight standard solutions were also prepared in such a way that there was as wide a variation as possible in the compositions (see Table III*a*).

The eight standard solutions were then examined in the X-ray fluorescent spectrometer, the excitation conditions being standardised on 40 kV., 24 mA. Fe, Co, Ni, Cr and Mo were counted in turn, and the number of counts/sec. plotted against percentage weight of the element. In this way the calibration graphs seen in Figs. 1–5 were obtained. It was considered unnecessary to make corrections for the background and total counts/sec. are plotted in the graphs.

The four residues in solution were then counted and from the counts/sec. the percentage weight of each element was interpolated by reference to the calibration graphs. The analyses obtained are shown in Table II for direct comparison with the values obtained by the conventional wet chemical method. A second sample of the first steel (from the same bulk material) was also analysed to determine the repeatability of the method, and the two results are shown as (*a*) and (*b*) in Table II.

Analysis of Residues from Two Cr-Mo-V Steels

Two residues were obtained from a 0·44% C, 5·21% Cr, 1·23% Mo, 0·73% V steel and a 0·43% C, 5·04% Cr, 2·40% Mo and 0·47% V steel, respectively. It was necessary in this case to obtain a calibration graph for vanadium. Five synthetic solutions were prepared, the analyses being shown in Table III*b*. These have the same proportions of Cr, Fe, Co and Mo as solutions 1, 3, 4, 5 and 6 of Table IIIa, the only difference being the replacement of Ni by V. All the elements were counted and Cr, Fe, Co and Mo were found to plot on the same calibration graphs as before; i.e. Figs. 1 to 5 apply whether vanadium is present in the solution or not. A calibration graph was also obtained for vanadium: this is shown in Fig. 6.

The two residues were chemically analysed by the conventional wet method and by X-ray fluorescence, and the results are shown in Table IV for direct comparison. As expected, the Mo : V ratio is higher in the second residue.

TABLE IV.—ANALYSES OF RESIDUES FROM TWO Cr-Mo-V STEELS

Steel No.	Method	Cr%	Mo%	V%	Fe%
1	X-ray	34·0	6·8	7·0	28·7
	Chemical	35·2	6·3	6·5	29·0
2	X-ray	32·5	13·5	4·0	27·0
	Chemical	33·1	12·9	3·9	27·9

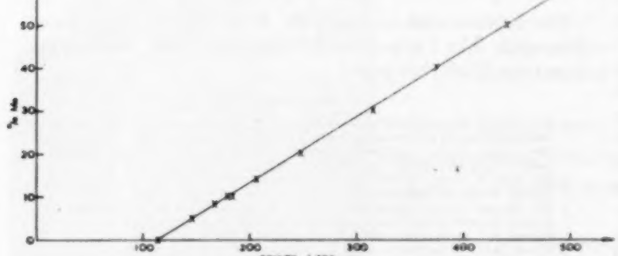


Fig. 5.—Calibration curve for molybdenum.

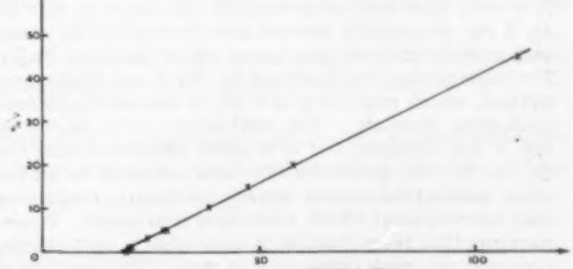


Fig. 6.—Calibration curve for vanadium.

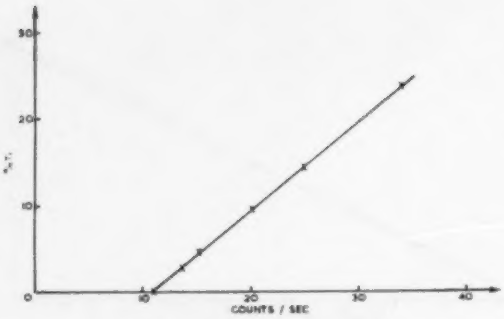


Fig. 7.—Calibration curve for titanium.

Analysis of Residue ($M_{23}C_6$) from a 12% Cr Steel

A residue was obtained from a 12% Cr, 2½% Mo, 6% Co, ½% V steel after ageing for 1,000 hours at 550° C. It was analysed for Cr, Mo, Co, V and Fe by the present method and by the usual wet method, and the results are shown in Table V. It is seen that neither Co nor V (in this case) enters into solid solution in the $M_{23}C_6$ to any appreciable extent, and that it may be given a formula approximately $Fe_6Cr_{13}Mo_4C_6$.

Two Applications of the Method

A Series of 12% Cr Steels

Residues were obtained from samples heat treated for 1 hour at 650° C. from six 12% Cr steels, the analyses of which are shown in Table VI. A small amount in each case was photographed in a Guinier-type focusing camera. The phases present are also incorporated in Table VI. M_2X is a hexagonal carbide based in this case on Mo_2C. Its occurrence and its metallurgical significance is discussed in detail in reference 7.

The six residues were also analysed by the method already described, with the results set out in Table VII. The following interesting conclusions were drawn from these analyses.

(*i*) Neither Ni nor Co appears to enter into solid solution with $M_{23}C_6$.

(*ii*) Vanadium on the other hand does so to a considerable extent, the $M_{23}C_6$ from the 2% V steel containing over 20% V.

Analysis of Chi Phase in the Fe-Cr-Ni-Ti System

The discovery of a *Chi* phase in Fe-Cr-Ni-Ti alloys has been described [8]. A residue was extracted from an alloy containing C 0·013%, Mn 1·29%, Cr 21·8%, Ni 4·96%, Ti 8·10% after heat treatment for 120 hours at 850° C. An X-ray photograph showed it to be mainly *Chi* phase with a small amount of a Laves phase based on Fe_2Ti. The same residue was analysed by the X-ray fluorescent method, which required a new set of standard solutions containing titanium. The calibration curve shown in Fig. 7 was obtained and it is again significant that the Fe, Cr, Ni, etc., points for the same solutions lie on the other calibration curves already obtained, confirming that inter-element effects have been eliminated. It also confirms that re-calibration is unnecessary, spot checks, however, are made from time to time. The analysis of the residue gave 47·5% Fe, 11·5% Cr, 2·1% Ni and 16·5% Ti.

TABLE V.—ANALYSIS OF RESIDUE ($M_{23}C_6$) FROM A 12% CR, 2½% MO, 6% CO STEEL

Steel No.	Method	Cr%	Mo%	Co%	V%	Fe%
1	X-ray	37·8	21·0	1·2	1·0	18·5
	Chemical	38·5	22·0	1·7	0·9	20·1

TABLE VI.—ANALYSES OF 12% CR STEEL AND PHASES PRESENT AFTER 1 HOUR AT 650° C.

C%	Mn%	Si%	Ni%	Cr%	Mo%	Co%	V%	Phases Present
0·105	0·69	0·29	2·98	12·01	3·07	—	—	$M_{23}C_6 + M_2X$
0·098	0·48	0·27	4·02	11·93	3·20	—	—	$M_{23}C_6 + M_2X$
0·104	0·50	0·19	—	12·03	—	5·12	0·53	$M_{23}C_6$
0·106	0·75	0·37	2·16	12·25	—	5·10	0·55	$M_{23}C_6$
0·097	0·62	0·31	2·05	12·39	—	5·00	1·00	$M_{23}C_6$
0·094	0·67	0·32	2·09	12·05	—	5·20	1·90	$M_{23}C_6$

TABLE VII.—ANALYSES OF RESIDUES FROM THE SIX 12% CR STEELS

Fe%	Cr%	Ni%	Co%	Mo%	V%
5·8	38·7	0·2	—	21·0	—
5·5	41·4	0·2	—	21·5	—
18·0	51·4	—	0·5	—	3·4
15·0	59·3	1·3	0·4	—	3·2
17·0	53·0	1·1	0·4	—	8·0
11·8	40·3	0·7	0·1	—	20·2

Discussion of Results

(1) It has been established that the analysis, by X-ray fluorescence, of residues obtained electrolytically from steels is feasible. In view of the small amount of sample used (0·1 g.) the results are considered to be very good. It might be possible, with experience, to improve the accuracy, but even as it stands the method can contribute very valuable information in addition to that already being obtained by established X-ray diffraction and electron microscope techniques.

(2) The method is not limited to electrolytic residues but can be used for any material. At the moment the application has been confined to the analysis of Fe, Co, Ni, Mo, Cr, V and Ti. Cu, Zn, Mn could readily be included in this list and others such as Nb with more difficulty. The lighter elements such as Al and Si are not being considered at present.

(3) The method is quick, and can be performed by an unqualified assistant. Once the calibration curves have been obtained about six powders a day can be completely analysed.

(4) The analyses reported here have been carried out on 0·1 g. of sample in 25 ml. Smaller amounts, down to approximately 0·05 g., can be analysed by dissolving in a proportionally smaller volume of acid, but the accuracy of the analysis will be somewhat poorer. The ultimate aim should be to analyse even smaller amounts, including possibly electron microscope replicas.

Acknowledgment

The authors wish to thank Mr. F. H. Saniter, Director of Research, The United Steel Companies, Ltd., for permission to publish this paper.

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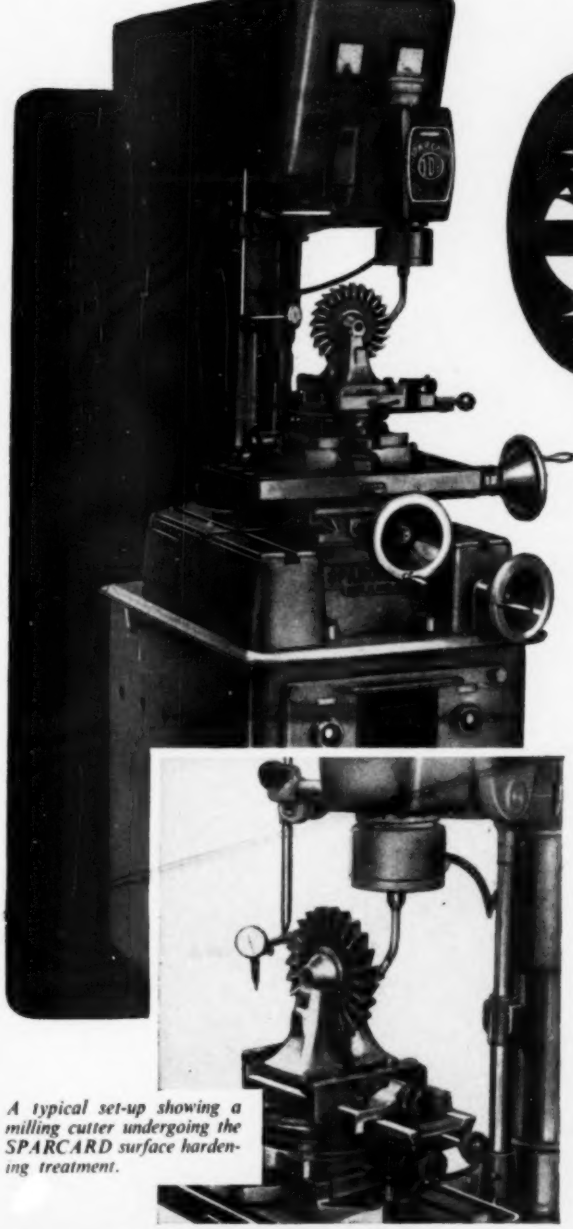

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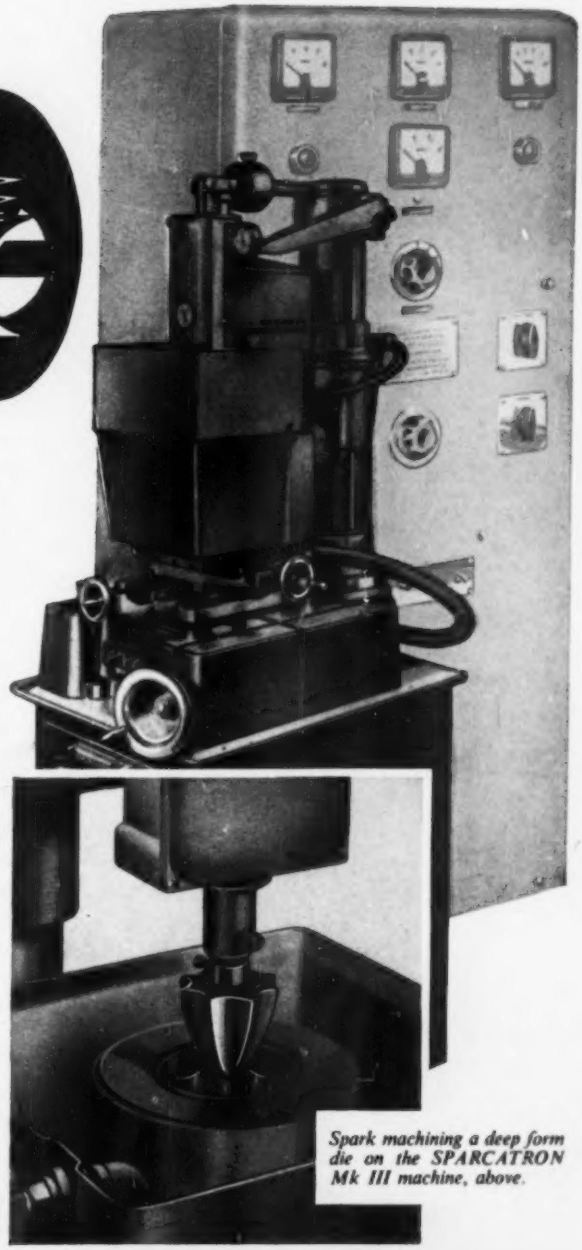

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